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Analyses of Grab Samples from Fixed-Bed Coal Gasification Processes

Radian Corp, Austin, Tex

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ANALYSES OF GRAB SAMPLES FROM FIXED-BED COAL GASIFICATION PROCESSES

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Program Report

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ABSTRACT

This report describes the results of an analytical screening study of selected effluent samples from operating coal gasification units. This work was done to aid in planning for future more comprehensive environmental test programs which will be conducted at gasification facilities located both in this country and abroad. A secondary objective of this work was to evaluate EPA's phased approach to environmental testing of coal gasification facilities.

Grab samples from five different commercial-scale gasifiers and one pilot-scale gasifier were obtained for this study. These samples included tar, process condensate, gasifier ash and cyclone dust. All of the gasifiers sampled were single-stage, fixed-bed, atmospheric pressure, air-blown units; however, the coal feed materials consumed by those gasifiers included both bituminous and anthracite coals. The analyses which were performed were intended to identify, in a very preliminary manner, the classes of compounds present in each sample which warrant further attention.

A number of significant conclusions are drawn from the results of this screening study. These conclusions relate to: a) the classes and concentrations of components found in specific samples, b) the effects of feedstock and process operating condition changes upon the levels of key components detected in those samples, and c) modifications/additions to suggested EPA procedures which will help to insure that the maximum amount of useful environmental information is obtained from future programs.

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SECTION 1.0
INTRODUCTION

This report describes the results of some preliminary analyses of grab samples of selected effluents from several coal gasification plants. This work was done as part of an EPA sponsored program (EPA contract No. 68-02-2147) whose overall objective is to provide a comprehensive environmental assessment of low-Btu coal gasification and utilization processes. The purpose of this specific study was to gain an insight into the nature of the samples that will be encountered in an ongoing test program and, also, to gain experience with the analytical methods proposed for use in the program.

The work reported here includes analyses as defined by the Environmental Protection Agency - Industrial Environmental Research Laboratory/Research Triangle Park (EPA-IERL/RTP) Procedures Manual[1] for a Level 1 environmental assessment, plus some additional characterization of the samples. The samples studied, which were taken from several sites, are identified in Table 1-1. Specific sample point locations at each site are identified in Section 2.0.

Major emphasis was placed on samples from Site A, since that facility had already been selected as a priority site for detailed testing at a later date. Work performed on samples from other sites was done primarily to complement the work on the Site A samples, thus providing a basis for comparing potential test sites. Another objective was to gain some concept of the effects that might be expected from differences in plant design and feedstock type.

Table 1-1. SAMPLES COLLECTED FOR PRELIMINARY SCREENING

Site*	Feed coal type	Cyclone bottom dust	Tar	Gasifier bottom ash	Process condensate	Gas duct dust
A	Bituminous	X	X	X	X	-
B1	Anthracite	X	-	-	-	-
B2	Anthracite	-	-	-	-	X
B3	Anthracite	X	-	-	-	-
C1	Bituminous	X	-	-	-	X
C2	Bituminous	-	X	-	X	-

* All sites utilize single-stage, fixed-bed, air-blown, atmospheric pressure gasifiers

This report consists of six sections. Following the Introduction, Section 2.0 describes the processes used and the samples collected at each site. Section 3.0 describes the analyses performed and the methods used. The analytical results are presented in Section 4.0. A discussion of these results follows in Section 5.0 while Section 6.0 provides a summary of the conclusions and recommendations which resulted from this work.

SECTION 2.0
SAMPLE SOURCES AND PROCESS DESCRIPTIONS

The samples examined in this study included quench water (process condensate), tar, ash, and cyclone dust from coal gasification plants at five different sites. All of these plants employ fixed-bed, single-stage gasifiers operated at atmospheric pressure. The following text provides a description of the processes used at each site and identifies the sources of the respective samples. Since major emphasis was placed on the samples from Site A, it is used as the descriptive model.

2.1 PROCESS DESCRIPTION - SITE A

The coal gasification unit at Site A produces low-Btu gas from bituminous coal. The product gas is used as a combustion fuel in process furnaces. The plant contains a series of gasifiers coupled to a gas main which feeds a gas scrubbing system. The number of gasifiers operating at any given time is determined by process fuel needs. At the time of sampling, two gasifiers were in operation.

A block diagram of the Site A facility is shown in Figure 2-1. The numbers on the diagram identify the sources of the respective grab samples. In the following text each of the processing operations used at Site A is described.

2.1.1 Coal Handling

The coal handling operation at Site A consists of: 1) delivery/storage of presized bituminous coal, 2) conveying,

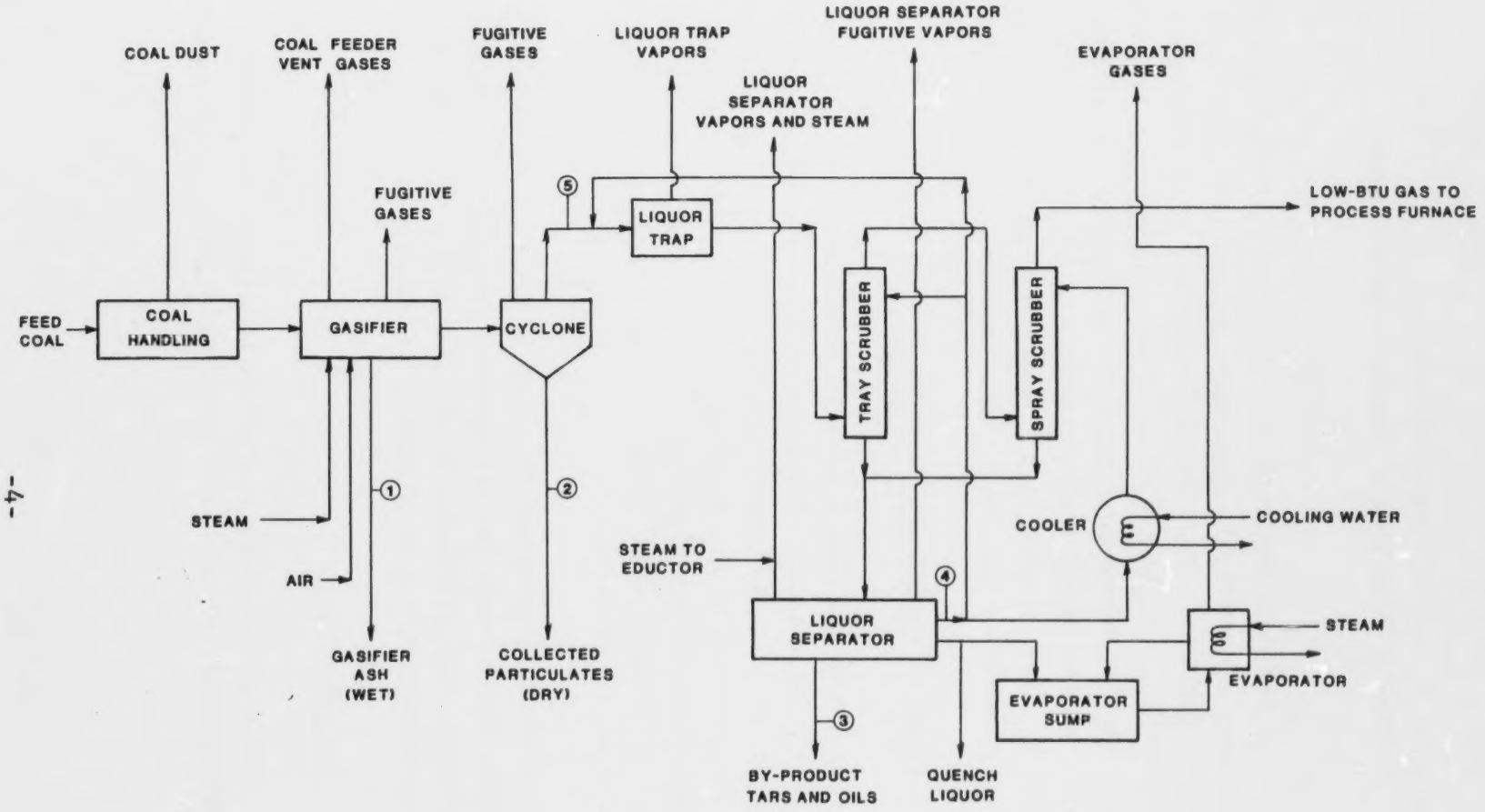


Figure 2-1. GASIFICATION UNIT PROCESS FLOW DIAGRAM - SITE A

and c) storing the coal in the gasifier feed hoppers. The main environmental problem associated with this operation is the generation of coal dust.

2.1.2 Coal Gasification

The gas producers at Site A are atmospheric, single-stage, fixed-bed, air-blown gasifiers. The coal feedstock enters the top of each gasifier through a barrel valve and is spread across the bed by a distribution arm. Steam and air are introduced into the bottom of the gasifier and pass through a grate into the gasification zone. The grate supports the bed and distributes the feed gases. Ash from the gasifier is collected in a water sealed ash pan and removed from the unit using an ash plow. (The ash sample for this study was taken from the ash pan following a recent operation of the ash plow.) The raw product gas at Site A leaves the top of the gasifier at 840-950°K (1050-1250°F).

2.1.3 Gas Purification

Particulates are removed from the hot product gas at Site A in a refractory-lined cyclone that operates at a temperature which is slightly lower than the gasifer overhead temperature. Each gasifier is equipped with its own cyclone. Trapped particulates which collect at the bottom of each cyclone are dumped periodically. The cyclone dust sample gathered for this study was taken from a batch of recently dumped dust which was allowed to cool in air.

The gas leaving the cyclones is quenched first by a series of sprays located inside the gas collection main. It then passes through two scrubbers in series where most of the

residual tars, oils, and particulates are removed as the gas is cooled to approximately 325°K (125°F). The effluent scrubbing liquor is sent to a tar-liquor separator.

In the liquor separator, tars and oils are separated from the aqueous condensate. The tars and oils are used as a combustion fuel. Most of the water is recirculated to the quenching operations. Any excess water that accumulates in the system during the gasification process is pumped to an evaporator sump. When the sump reaches a specified level, the excess condensate is sent to an evaporator where it is evaporated and vented to the atmosphere. The samples of Site A tar and quench liquor used in this study were taken from the separator system.

2.2 PROCESS DESCRIPTION - SITES B1-3

Sites B1, B2, and B3 all use fixed-bed gasifiers which are similar to the units used at Site A. The hot product gas at all "B" sites is used directly as a combustion fuel after the large particulates in the product gas are removed in a hot cyclone. Since anthracite coal is used as a feedstock at all "B" sites, these systems produce essentially no tar.

Two of the "B" site particulate samples were taken from the bottom of the cyclone and another sample from the exit of the product gas main. These sample points correspond to points 2 and 5, respectively, in Figure 2-1.

2.3 PROCESS DESCRIPTION - SITE C

The gasifiers at Site C are fixed-bed units which are similar in principle to those described previously. Samples were taken from two different systems. One system (Site C1)

consisted of a commercial scale gasifier and cyclone coupled to a flare by a long run of insulated duct. One dust sample from this unit was taken from the gas duct between the cyclone and the flare. A cyclone dust sample from this unit was also taken from the bottom of the cyclone (at point 2, as identified in Figure 2-1). The other system which was sampled at Site C was a small (two foot diameter gasifier) process development unit that included a wash box (a single-stage scrubbing column) and an electrostatic precipitator. A schematic diagram of this system is shown in Figure 2-2. The process development unit tar and wash box water samples which were studied were "old" samples which had been previously obtained (approximately six months prior to the time that the analyses reported here were performed) from sample points A and B (as shown in Figure 2-2).

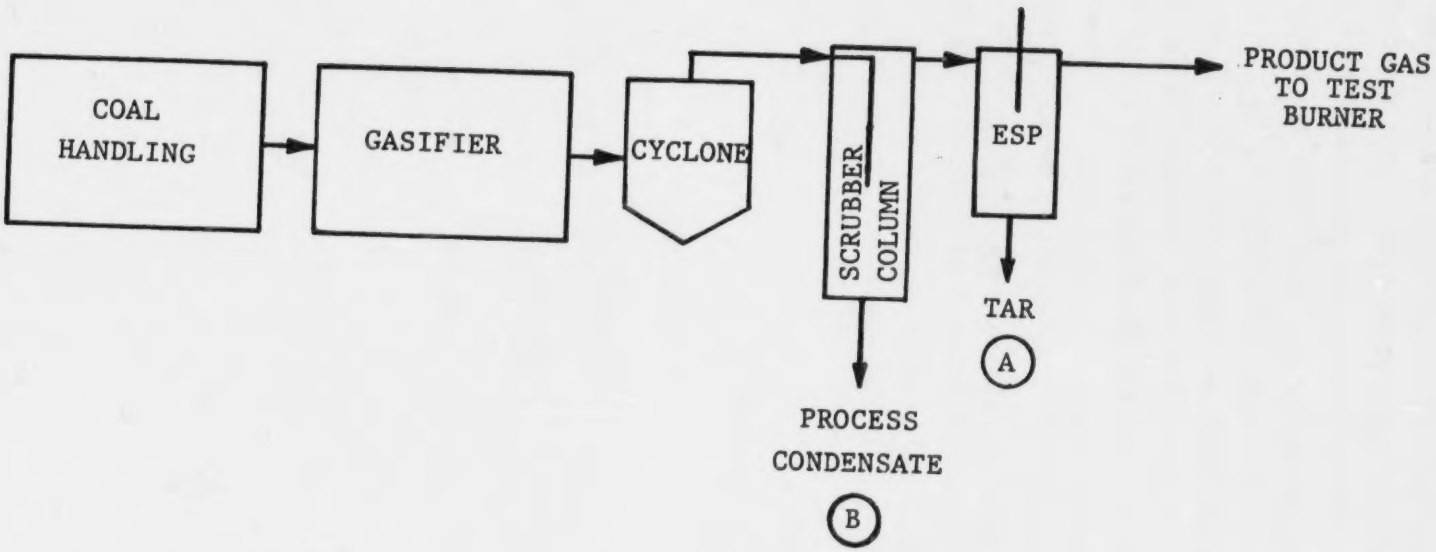


Figure 2-2. SCHEMATIC DIAGRAM OF THE SITE C2 PROCESS DEVELOPMENT UNIT

SECTION 3.0
ANALYSES PERFORMED

This section of the report provides a description of the analytical approach used in this screening study. Section 3.1 describes the analysis strategy defined by Level 1 procedures and Section 3.2 spells out the analyses actually performed on each sample.

3.1 OVERALL ANALYTICAL STRATEGY

The basic analytical strategy used for this study is illustrated in Figure 3-1. This strategy follows the procedures recommended for a Level 1 environmental assessment[1].

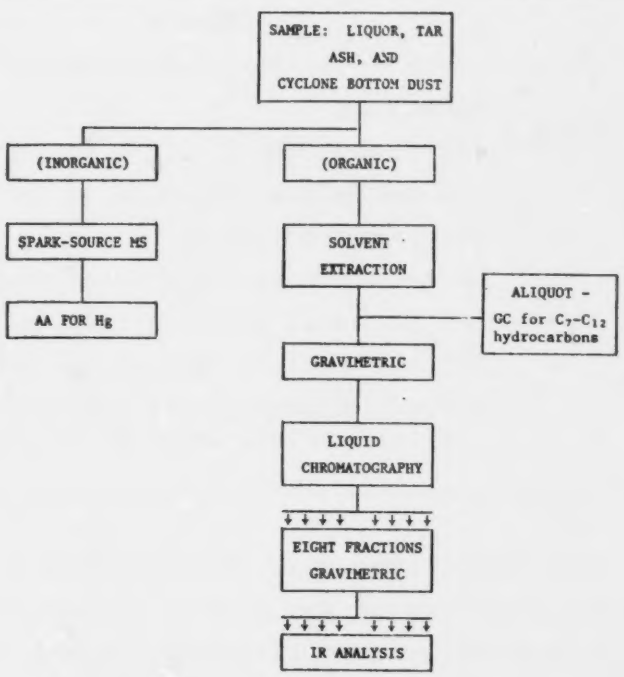


Figure 3-1. SIMPLIFIED DIAGRAM OF ANALYSIS STRATEGY DEFINED BY LEVEL 1 PROCEDURES

3.1.1 Organic Analysis

Extraction and Liquid Chromatography (LC) -

A standardized scheme, as defined by the EPA Level 1 procedure, was applied to all samples. By this procedure, the sample was extracted with methylene chloride and the mass of the extracted material determined. Prior to concentrating the sample extract, however, a 1 mℓ portion of the solvent/extract mixture was reserved for analysis by gas chromatography (GC). The concentrated extract was then separated into eight fractions by liquid chromatography using the elution sequence shown in Table 3-1.

Table 3-1. LEVEL 1 ELUTION SEQUENCE FOR LC SEPARATION OF EXTRACTED ORGANICS

Fraction	Eluent
1	n-Pentane
2	20% methylene chloride in pentane
3	50% methylene chloride in pentane
4	methylene chloride
5	5% methanol in methylene chloride
6	20% methanol in methylene chloride
7	50% methanol in methylene chloride
8	5/70/30 concentrated HCl/methanol/ methylene chloride

Samples were evaporated onto the column packing in order to effect a dry transfer of the sample to the column. The carrier solvent was removed by evaporation and after the mass

of the extract was determined, the respective fractions were examined by infrared (IR) spectrometry.

Gas Chromatographic Analysis -

The gas chromatographic determination of volatile organics in the sample extract, reported as C_7-C_{12} hydrocarbons, was accomplished using a modification of Level 1 procedures. A 3% OV-101 column was used at 50°C for four minutes, followed by a temperature program from 50°C to 150°C at 8°C per minute. The hold time at 150°C was eight minutes. All other conditions were those specified by Level 1.

A mixture of hydrocarbon standards was used to calibrate the instrument for retention times and quantitative response. The results from each sample are reported as specified by Level 1 procedures. For example, everything eluting in the boiling range of 90° to 110°C is reported as C_7 hydrocarbons.

Characterization of Organics by IR Spectrometry -

The infrared spectrum, between 4000-400 cm^{-1}, was obtained for each of the concentrated extracts and for the respective chromatographic fractions from each extract by using the following procedure. An appropriate portion of each sample was taken up in a small quantity of methylene chloride and deposited on a KBr-plate. The plate was placed under an infrared lamp and purged with a gentle stream of dry nitrogen until the solvent evaporated*. In some cases the neat sample flowed, as a melt, over the plate. Samples which showed evidence of strong water bands were transferred to an IrTran II window and

---

* Footnote. Since the Level 1 Procedures Manual[1] does not define this procedure explicitly, this commonly accepted practice was followed.

scanned again. IR spectra were obtained on the following matrices:

- Extract of quench liquor and fractions of quench liquor extract
- Extract of tar and fractions of tar extract
- Extract of cyclone bottom dust

3.1.2 Inorganic Analyses (Quench Liquor, Tar and Cyclone Dust)

A representative portion of each sample was submitted to Commercial Testing and Engineering Company, 228 North LaSalle Street, Chicago, Illinois 60601, for elemental analysis by spark-source mass spectrometry (SSMS) and, in the case of mercury, atomic absorption spectrometry (AAS).

3.2 SPECIFIC SAMPLES ANALYZED AND METHODS USED

A summary of the samples analyzed is contained in Table 3-2. The methods used to analyze the respective samples are described below.

3.2.1 Characterization of Quench Liquor

Organic Analyses -

A 100-mℓ portion of the quench liquor was extracted with methylene chloride. Four equal volume extractions were used*. As illustrated diagrammatically in Figure 3-1, a 1-mℓ portion of the combined extract was reserved for analysis by

---

*Footnote. The Level 1 Procedure Manual[1] recommends that three 500 mℓ extractions of a 10 liter sample be used. The organic content of this sample did not justify the use of such proportions. Further, a more favorable partition ratio was sought for the components involved - see Section 5.5.1 of this report.

TABLE 3-2. ANALYSES PERFORMED ON VARIOUS SAMPLES

	Extractables	IR of Extract	GC of Extract	LC of 8 Fractions	IR of Fractions	Trace Elements	Optical Microscopy	Ash	Bulk Density	Moisture	CHNS Elemental Analyses	Screen Test
Site A Samples												
Quench liquor	X	X	X	X	X	X	-	-	-	-	-	-
Tar	X	X	X	X	X	X	-	-	-	-	-	-
Cyclone dust	X	X	X	X	X	X	X	X	X	X	X	X
Ash	X	X	-	-	-	X	-	-	-	-	-	-
(Tar residue)	-	-	-	-	-	-	X	-	-	-	-	-
Site B Samples												
Cyclone dust	-	-	-	-	-	-	X	X	X	X	-	X
Duct dust	-	-	-	-	-	-	X	X	X	X	-	-
Site C Samples												
Quench water	X	X	-	-	-	-	-	-	-	-	-	-
Tar	X	X	-	X	X	-	-	-	-	-	-	-
Cyclone dust	-	-	-	-	-	-	X	X	X	-	-	X
Duct dust	-	-	-	-	-	-	X	X	X	-	-	-
(Tar residue)	-	-	-	-	-	-	X	-	-	-	-	-
	Level 1 Analyses							Additional Characterization				

GC. The balance of the extract was concentrated by evaporation and weighed. The concentrate was then transferred to a silica gel column and separated into eight fractions using the Level 1 elution sequence shown in Table 3-1. The respective fractions were then weighed and examined by IR spectrometry.

Following extraction by the Level 1 procedure, the liquor was then extracted with two 40-mℓ volumes of diethyl ether. The extracted liquor was then acidified with HCl to a pH of 1 and extracted with two additional volumes of diethyl ether. The respective extracts were concentrated by evaporation and weighed. These additional extractions were done to determine whether or not the extractable organics were completely removed by the Level 1 extraction procedure.

Inorganic Analyses -

A representative portion of each sample was submitted to Commercial Testing and Engineering Company for elemental analysis by SSMS and AAS.

3.2.2 Characterization of Tar

Organic Analyses -

A 0.50209 gram sample of tar was extracted for 24 hours in a Soxhlet extractor with 200 mℓ of CH_2Cl_2. A 1-mℓ portion of the extract was reserved for GC analysis by the method described previously. The balance was concentrated by evaporation and weighed, after which the extract was transferred to a silica gel column and separated into eight fractions by the Level 1 procedure described previously. The respective fractions were then weighed and examined by infrared spectrometry.

The residue in the extraction thimble was extracted with tetrahydrofuran (THF) to check for the presence of materials that are insoluble in methylene chloride.

Determination of Residue Insoluble in Methylene Chloride - A 10.12 gram sample of tar was dissolved in methylene chloride and filtered through a medium-frit filter. The filtrate was then filtered through Whatman No. 42 filter paper to isolate submicron particles which may have passed through the medium-frit filter. The weight of each residue was determined, after which the residues were examined by optical microscopy.

Inorganic Analyses -

A representative portion of each sample was submitted to Commercial Testing and Engineering Company for elemental analysis by SSMS and AAS.

3.2.3 Characterization of Cyclone Bottom Dust

Organic Analyses -

A 100-gram sample of cyclone bottom dust was extracted for 24 hours in a Soxhlet extractor with 200 mℓ of methylene chloride. A 1-mℓ portion of the extract was reserved for GC analysis. The balance was concentrated by evaporation and weighed, after which it was subjected to the chromatographic and IR procedures described previously.

Elemental Analyses for Major Elements -

Elemental analyses for C, H, N, and S were obtained from Galbraith Laboratories, Knoxville, Tennessee.

Inorganic Analyses -

A representative portion of each sample was submitted to Commercial Testing and Engineering Company for elemental analysis by SSMS and AAS.

Optical Microscopy -

Cyclone bottom ash was examined by optical microscopy at 20x and 100x magnification. Photomicrographs were prepared to document particle shape and relative particle size distribution. Micrographs of the cyclone dusts from the sources identified in Table 1-1 were then compared visually.

3.2.4 Characterization of Gasifier Ash

Organic Analyses -

A 50.3 gram sample of gasifier ash was extracted for 24 hours in a Soxhlet extractor with 200 mℓ of methylene chloride. A 1-mℓ portion of the extract was reserved for GC analysis. The balance was concentrated by evaporation and weighed. (Since the mass of the organics extracted was less than the 8 mg minimum specified by the Level 1 procedure, no chromatographic separation was made.)

Inorganic Analyses -

A representative portion of each sample was submitted to Commercial Testing and Engineering Company for elemental analysis by SSMS and AAS.

SECTION 4.0
RESULTS

The results from the analyses of the various samples are reported in this section, along with a discussion of some of the more significant results. Analytical flow diagrams are provided to aid in following the presentations of data.

4.1 ANALYSIS OF SITE A QUENCH LIQUOR

The analytical flow diagram for the analysis of Site A quench liquor is shown in Figure 4-1.

4.1.1 Organic Analyses

The results of the extraction test on Site A quench liquor are summarized in the following table.

Table 4-1. ORGANICS EXTRACTED FROM SITE A QUENCH LIQUOR

Step		mg/ℓ	% of total extracted
1	Methylene Chloride Extractable	2231	57.7
2	Ether Extractable[a] at pH 7	1457	37.6
3	Ether Extractable[b] at pH 1	177	4.7
	TOTAL EXTRACTED	3865	100

[a]Following Step 1

[b]Following Steps 1 and 2

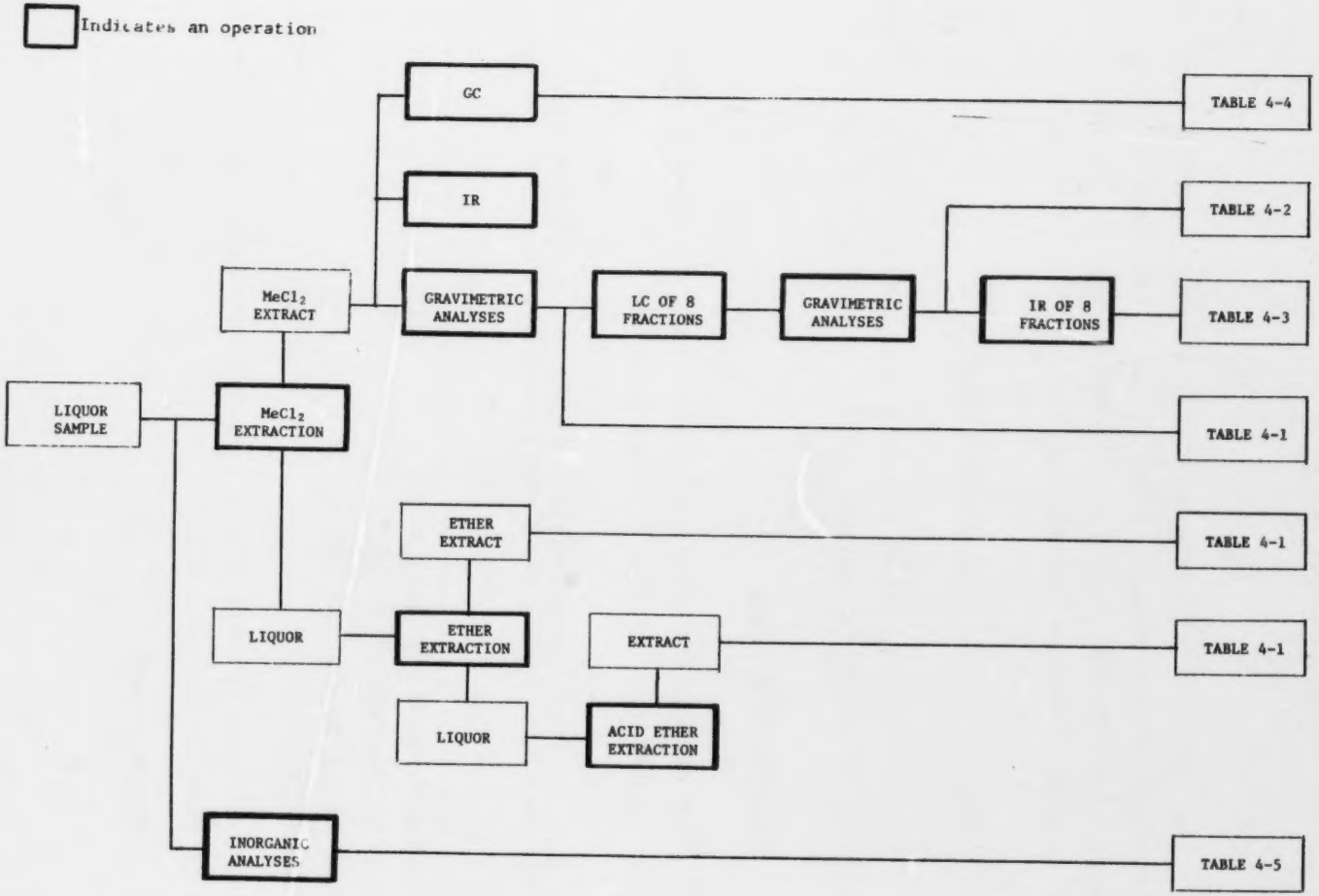


Figure 4-1. ANALYTICAL FLOW DIAGRAM FOR QUENCH LIQUOR SAMPLE FROM SITE A

Liquid Chromatographic Separation -

The mass distribution of the methylene chloride extract, as determined by LC, is shown in Table 4-2. Most of the extract was eluted in Fraction 6 (solvent = 20% methanol in methylene chloride). The IR spectrum of this fraction is very similar to that of phenol.

Table 4-2. LC MASS DISTRIBUTION OF SITE A QUENCH LIQUOR EXTRACT

Fraction number	mg/ℓ	% of amount eluted	% of amount loaded
1	0.3	0.01	0.01
2	1.8	0.09	0.08
3	5.0	0.24	0.22
4	17.0	0.81	0.76
5	163.0	7.77	7.31
6	1655.0	78.92	74.18
7	69.0	3.29	3.09
8	186.0	8.87	8.34
Total	2097.1	100.00%	93.99%

Total Recovered	2097 mg/ℓ
Total Loaded	2231 mg/ℓ
% Recovered	94.0 %

IR Spectral Characterization -

The following table contains a listing of the functional group assignments on each fraction as indicated by the IR spectra.

Table 4-3. SPECTRAL COMPOSITION OF SITE A QUENCH LIQUOR EXTRACT

Fraction Number	% of Total Extract	Functional Groups Present
1	0.01	Aliphatic hydrocarbons
2	0.08	Alkyl-aryl hydrocarbons
3	0.22	Alkyl-aryl hydrocarbons, trace carbonyl, possible polycyclics or multi-substituted aromatics
4	0.76	Alkyl-aryl hydrocarbons, possible polycyclics, -OH present (possibly atmospheric moisture)
5	7.31	Phenol + alkyl/dialkyl phenols
6	74.18	Principally Phenol + other phenols
7	3.09	Phenols
8	8.34	Alkyl-aryl CH-stretch, OH, $-\overset{\vert}{\underset{\vert}{C}}=O$, Methyl Bending Vibration, Primary OH, possible inorganic sulfur + other ionic compounds

GC Analyses -

The results of the GC analysis of the site A quench liquor extract are shown in Table 4-4. The concentration values shown are based on calibration with an external standard. The material in the C_7 range is believed to originate with the extracting solvent since it is inconceivable that significant levels of C_7's could be present with no detectable quantities of either C_8's or C_9's.

Table 4-4. VOLATILE ORGANICS IN SITE A QUENCH LIQUOR EXTRACT -DETERMINED BY LEVEL 1 GC ANALYSIS

Range	Projected BP (°C)	ppm wt. in Quench Liquor
C_7	90-110	260
C_8	110-140	0
C_9	140-160	0
C_{10}	160-180	2544
C_{11}	180-200	2766
C_{12}	200-220	917
C_{12}	>220	800

4.1.2 Inorganic Analyses

The following table contains the results of the elemental analyses of site A quench water. All values except mercury were determined by SSMS. Attention is called to the 4 µg/ℓ value for selenium.

Table 4-5. INORGANIC ANALYSIS – TRACE ELEMENTS IN SITE A QUENCH LIQUOR

Element	µg/ℓ	Element	µg/ℓ
Lead	0.04	Gallium	0.006
Mercury*	0.007	Zinc	0.07
Neodymium	≤0.01	Copper	0.1
Praseodymium	0.005	Nickel	0.1
Cerium	0.01	Cobalt	≤0.008
Lanthanum	≤0.01	Iron	3
Barium	0.1	Manganese	0.03
Cesium	1	Chromium	0.03
Iodine	0.5	Vanadium	0.004
Antimony	0.1	Titanium	0.05
Tin	0.02	Scandium	≤0.006
Indium	Std	Calcium	MC
Cadmium	≤0.02	Potassium	MC
Molybdenum	0.06	Chlorine	0.3
Zirconium	0.01	Sulfur	MC
Yttrium	≤0.004	Phosphorus	MC
Strontium	0.2	Silicon	7
Rubidium	0.03	Aluminum	1
Bromine	0.2	Magnesium	2
Selenium	4	Sodium	MC
Arsenic	0.2	Fluorine	≈2
Germanium	≤0.02	Boron	2
		Lithium	0.2

*Flameless atomic absorption

MC = major component

NOTE: Any element not listed - concentration ≤0.004.
Carbon, hydrogen, nitrogen and oxygen are excluded from these analyses.

4.2 ANALYSIS OF SITE A TAR

The tar from site A was a black thermoplastic solid that was converted to a viscous liquid at 100°C. Its density was greater than water and it exhibited an odor similar to a commercial asphalt-based pitch (xylenols). The analytical flow diagram for this sample, which relates the procedural steps to the data reported below, is shown in Figure 4-2.

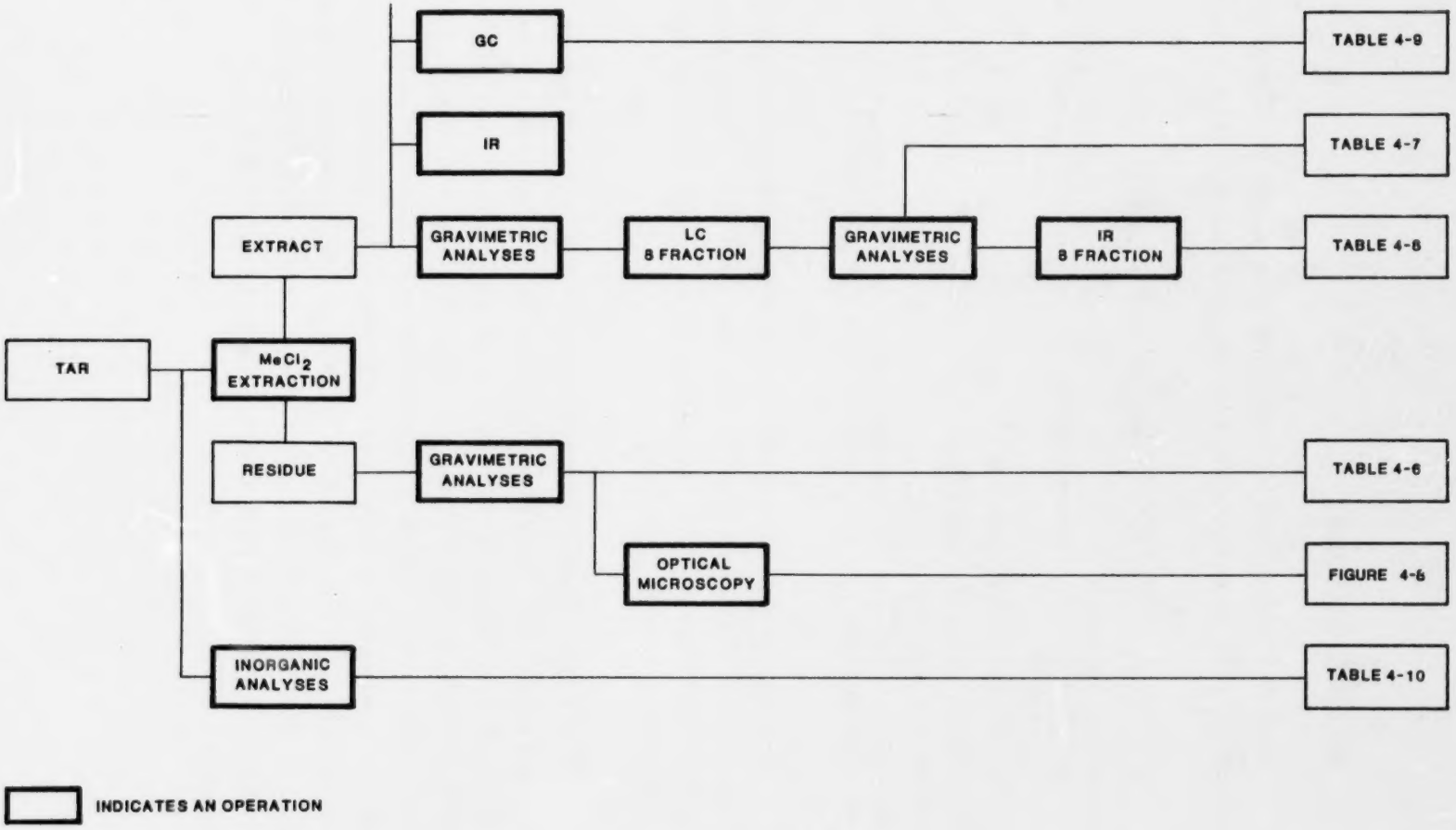


Figure 4-2. ANALYTICAL FLOW DIAGRAM FOR SITE A TAR

4.2.1 Organic Analyses

The following table contains the data obtained from the extraction and chromatographic separation of site A tar.

Table 4-6. ORGANIC EXTRACTABLES IN SITE A TAR

Extractables In Tar	grams	% of sample
Weight of Sample	0.50209	--
Weight of Extracted Organics	0.33583	66.9
Loss-on-Extraction/Evaporation	0.16625	33.1
Insoluble Residue	0.0200	4.0*

*3.9% on the medium frit + 0.1% on the filter paper

The insoluble residue microscopically was similar to the solids taken from the bottom of the cyclone. This material is discussed further in the sections devoted to particulate matter (Sections 4.3.3 and 5.1).

The significant loss-on-extraction and evaporation is not yet explained and requires further investigation. The THF extraction of the residue remaining in the thimble after extraction with methylene chloride accounted for only a minor part of the loss. Since the final evaporation was under an infrared lamp, it is conceivable that part of the loss resulted from the evaporation of some of the more volatile components. A thermometer placed under the lamp read 68°C. However, the sample may have reached a higher temperature since it probably had a lower reflectivity.

Fractionation by Column Chromatography -

The mass distribution of the chromatographic fractions of the Site A tar extract is shown in Table 4-7.

Table 4-7. LC MASS DISTRIBUTION OF SITE A TAR EXTRACT

Fraction	mg Eluted	mg/g Extract	% of Eluent
1	0.75	2.23	0.27
2	5.5	16.38	1.96
3	48.6	144.72	17.28
4	23.6	70.27	8.39
5	20.6	61.34	7.32
6	147.3	438.61	52.37
7	14.0	41.69	4.98
8	20.9	62.23	7.43
Total	281.25	837.48	100.00
Loss	54.58	162.52	--
Total Loaded on Column	335.83		

As with the condensate extract, the major portion of the tar extract eluted in Fraction 6. A comparison between the normalized mass distribution of the liquor and the tar extract is shown in Figure 4-3. Generally, the tar contains a broader distribution of materials, including greater amounts of less polar materials (those eluted in Fractions 1-4).

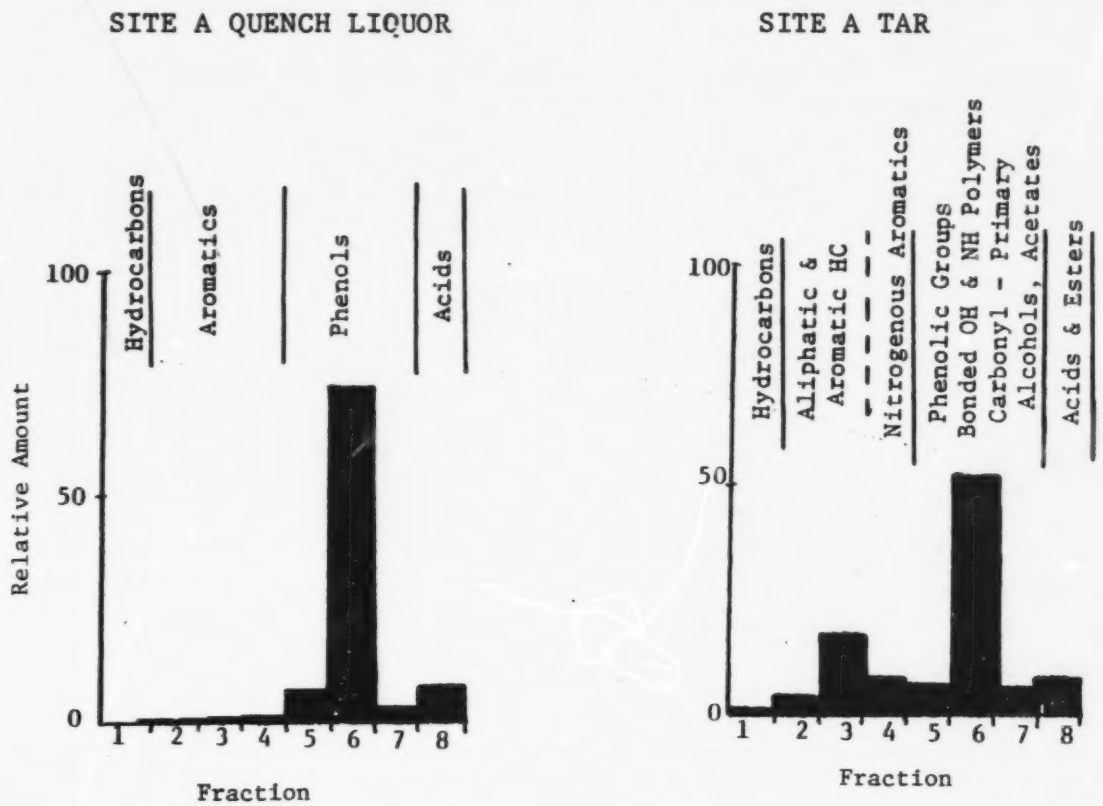


Figure 4-3. COMPARISON OF MASS DISTRIBUTIONS OF METHYLENE CHLORIDE EXTRACTABLES IN LC ELUENTS

IR Spectral Characterization -

The IR Spectra of the Site A tar extract and its respective fractions indicate that its composition is substantially different from that of the quench liquor extract. Although both spectra indicate the presence of phenolic groups, the tar spectra also indicate the presence of N-H with considerable intermolecular bonding. (This should not be a surprise since the tar is indeed an intermolecularly bonded thermoplastic polymer.) Table 4-8 shows a summary of the spectral interpretation of the respective Site A tar extract fractions.

Table 4-8. SPECTRAL COMPOSITION OF SITE A TAR EXTRACT

Fraction number	Composition (Functional groups present)	Relative percent in extract
1	Aliphatic hydrocarbons $(-CH_2-)>4$ some branched	0.27
2	Substituted aromatic with alkyl and branched alkyl substituents	1.96
3	Substituted aromatic with alkyl and branched alkyl substituents	17.28
4	Same as above plus NH stretch	8.39
5	Same as above but probably OH stretch, trace C=O	7.32
6	Bonded OH and NH stretch	52.37
7	Primary OH, water of hydration, CO ether, ester (acetate), split carbonyl (possible acid)	4.98
8	Split $>C=O$, (acid and ester), water of hydration, possible ester (acetate), aliphatic, methyl group	7.43

GC Analyses -

The results from the GC analyses of Site A tar extract are shown below. Analyses were performed on a 1 mℓ aliquot of a sample containing about 2.41 mg of tar extract/mℓ of methylene chloride. Results are reported in the form specified by Level 1 procedures.

Table 4-9. VOLATILE ORGANICS IN SITE A TAR BY LEVEL 1 GC ANALYSIS

Range	Projected BP(°C)	(mg/g tar)
C_7	90 to 110	3.0
C_8	110 to 140	0
C_9	140 to 160	0
C_{10}	160 to 180	18.2
C_{11}	180 to 200	56.7
C_{12}	200 to 220	72.5
$>C_{12}$	>220	307.5

The amount of material found in the C_7-C_{12} range constitutes 15.0% of the total sample. The total amount of material eluting (*i.e.*, C_7-C_{12} and $>C_{12}$) constitutes approximately 46% of the original amount of the tar sample.

4.2.2 Inorganic Analyses of Tar

The following table contains the results of the trace element analyses of the tar. All values except mercury were determined by SSMS. Mercury was determined by flameless AAS.

Any element not listed in the table, if present, was at a concentration of less than 0.2 ppm by weight. Other elements not reported include the major elements: carbon, hydrogen, nitrogen and oxygen.

Table 4-10. TRACE ELEMENTS IN SITE A TAR

Element	ppm wt.	Element	ppm wt.
Lead	10	Copper	3
Mercury	0.12	Nickel	5
Neodymium	0.6	Cobalt*	5
Praseodymium	0.3	Iron	120
Cerium	0.5	Manganese	0.9
Lanthanum	0.6	Chromium	3
Barium	27.0	Vanadium	0.8
Cesium	0.1	Titanium	29
Iodine	1	Scandium	0.7
Antimony	0.8	Calcium	630
Tin	0.9	Potassium	100
Molybdenum	1	Chlorine	6
Zirconium	0.7	Sulfur	520
Yttrium	$\leq$0.2	Phosphorus	17
Strontium	10	Silicon	170
Rubidium	0.2	Aluminum	25
Bromine	2	Magnesium	23
Selenium	3	Sodium	71
Arsenic	4	Fluorine	$\sim$22
Germanium	1	Boron	19
Gallium	8	Beryllium	0.1
Zinc	7	Lithium	4

*Heterogeneous

Attention is called to the concentration of selenium in the tar which is used as a supplementary fuel in a coal-fired power plant.

4.3 CHARACTERIZATION OF SITE A CYCLONE DUST

An analytical flow diagram for Site A cyclone dust is shown in Figure 4-4. This diagram relates the procedural steps followed to the data reported below. The dust from the bottom of the cyclone was observed to be a black, granular, free-flowing substance with a bulk density of 0.4 g/cc and a particle size ranging between <1 and 2000 microns, as determined by a screen test. The bulk of the material appeared to consist of particles in the 75-400μ range, as shown by the data in Table 4-11.

Table 4-11. RESULTS OF DRY SCREEN TEST OF SITE A CYCLONE DUST

Sieve No.	Particle size (dp [μ])	wt %	Cumulative
10	>2000	0.3	100.0
18	>1000	0.2	99.7
25	> 710	0.1	99.5
40	> 425	3.1	99.4
60	> 250	18.9	96.3
80	> 177	22.3	77.4
100	> 149	17.9	55.1
120	> 125	6.9	37.2
140	> 106	7.6	30.3
200	> 74	11.6	22.7
230	> 63	3.8	11.1
400	> 37	4.6	7.3
-400	< -37	2.7	2.7

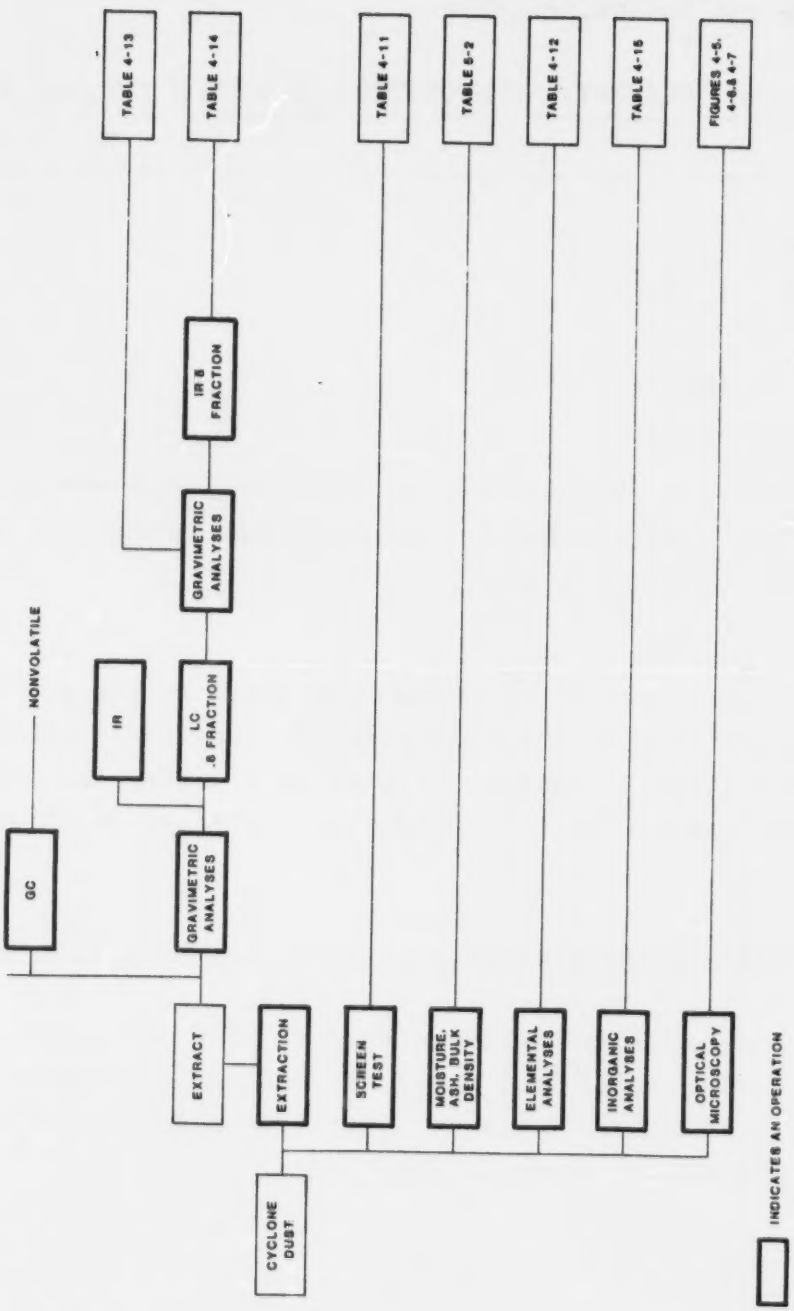


Figure 4-4. ANALYTICAL FLOW DIAGRAM FOR SITE A CYCLONE DUST

The elemental composition of the Site A cyclone dust is shown in the following table:

Table 4-12. ELEMENTAL COMPOSITION OF SITE A CYCLONE DUST

Element	Wt. Percent
Carbon	84.42
Hydrogen	1.62
Nitrogen	1.07
Sulfur*	0.67
Residue on Ignition (ROI)	10.19
Total (approximate)	98%

*Schoeniger flask method includes organic sulfur and soluble forms of inorganic sulfur.

Since some of the sulfur contained in the ROI fraction could also appear with the sulfur value, the total accounted for is only an indicative value. If used as a guide, the organic oxygen value appears to be in the vicinity of 2% as determined by difference.

4.3.1 Organic Analyses of Site A Cyclone Dust Extract (200g)

The methylene chloride extract from 100 grams of site A cyclone dust yielded 38.1 mg of organic residue representing 381 ppm or 0.38 mg/g. The mass distribution of the LC fractions from that extract is shown in Table 4-13.

Table 4-13. LC MASS DISTRIBUTION OF SITE A CYCLONE DUST EXTRACT

Fraction	mg Eluted	% of Eluted	ppm in dust
1	21.84	35.17	109
2	3.53	5.69	18
3	0.21	0.34	1
4	0.57	0.92	3
5	0.62	1.00	3
6	22.69	36.54	113
7	3.76	6.05	19
8	8.87	14.29	44
Total	62.09	100.00	310
Mass loaded	43.97*		
Excess recovered	18.12		

*Combined from two extractions

This cyclone dust sample was red hot when it was dropped from the cyclone into an open pan. Because of heat trasfer limitations, the dust cooled slowly. It is not known whether significant oxidation occurred during the cooling period, however, there was ample time for such oxidation to occur. This sampling procedure is not entirely faulty from an effluent characterization point of view, however, because in the process itself, the dust is dropped hot and allowed to cool in a deep hopper.

IR Spectral Characterization -

The organic extract from the Site A cyclone bottom dust is significantly different from both the tar extract and the quench liquor extract in that it is comparatively nonaromatic. The Site A cyclone dust extract contained a major paraffinic hydrocarbon fraction and a major ester-alcohol fraction.

Table 4-14 lists the major functional groups identified in the respective fractions.

Table 4-14. SPECTRAL COMPOSITION OF SITE A CYCLONE DUST EXTRACT

Fraction	% of Total Extract	Assignments
1	35.2	Paraffinic hydrocarbons, considerable branching
2	5.8	Paraffinic functional groups, traces of substituted aromatics
3	0.3	Split carbonyls, esters (formate or butryate), methyl, isopropyl and tributyl branching, primary alcohols)
4	0.9	
5	1.0	Aromatics, carbonyls, aliphatic hydrocarbons
6	36.5	Split carbonyls; methyl, isopropyl and tributyl branching; secondary alcohols; esters; possible 5C ring lactones; branched cyclic alcohols
7	14.3	(No assignment made)

GC Analyses -

The bulk extract from the Site A cyclone dust showed only one peak in the C_7 range with a concentration of 2.2 mg/g of dust. No other components were detected in the C_7-C_{12} range nor in the C_{12}+ range. This result is questionable since it would be expected that most of the organic species would be in the C_7+ range. Further investigations of the organics in cyclone dust extracts are suggested.

4.3.2 Inorganic Analyses

The levels of trace elements measured in the Site A cyclone dust are listed in Table 4-15. All values except mercury were obtained by SSMS. Mercury was obtained by flameless AAS.

4.3.3 Examination by Optical Microscopy

Microscopically, the Site A cyclone dust particles appeared to be primarily spherical in shape. They ranged in size from 15 to 90μ with many particles in the 50μ size range. Smaller particles appeared to be primarily fragments of broken spheres. Visually, the spheres appeared to be hollow. This observation is supported by the bulk density value of 0.4 g/cc. Many spheres appeared to have "blow holes" in their outer shells. These observations lead to an hypothesis that many of the particles were formed from materials which were suspended tar droplets initially. Around each suspended droplet, a carbonized skin may have formed from which volatile organics escaped, thus forming the perforated hollow spheres. Such particles could be quite buoyant in a flowing stream.

Photomicrographs -

A number of photomicrographs are included here to illustrate the observations described above. However, it must be recognized that a two-dimensional presentation cannot possibly convey the information obtained subjectively by direct examination. Superior detail can also be achieved with a scanning electron microscope, as illustrated by the two micrographs in Figure 4-5.

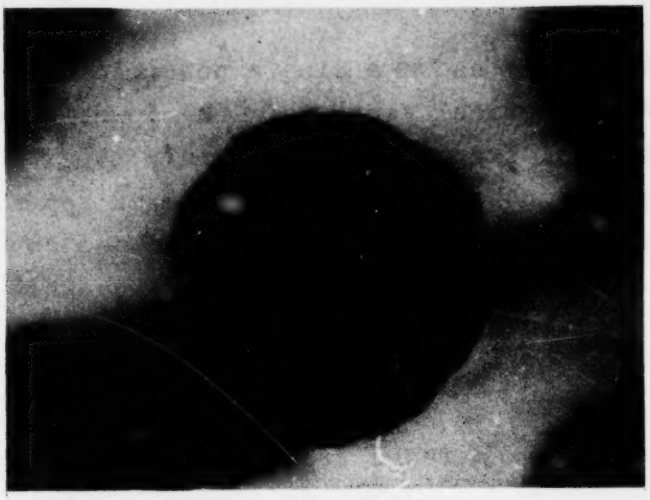
Table 4-15. TRACE ELEMENTS IN SITE A CYCLONE DUST

Element	ppm wt	Element	ppm wt
Bismuth	2	Arsenic	27
Lead	60	Germanium	5
Mercury*	0.01	Gallium	130
Terbium	9	Zinc	85
Gadolinium	2	Copper	130
Europium	1	Nickel	30
Samarium	9	Cobalt	16
Neodymium	21	Iron	MC
Praseodymium	5	Manganese	120
Cerium	45	Chromium	90
Lanthanum	45	Vanadium	100
Barium	460	Titanium	MC
Cesium	1	Scandium	12
Iodine	4	Calcium	MC
Antimony	8	Potassium	MC
Tin	2	Chlorine	720
Indium	Std	Sulfur	MC
Cadmium	<2	Phosphorus	MC
Silver	3	Silicon	MC
Molybdenum	14	Aluminum	MC
Niobium	12	Magnesium	MC
Zirconium	80	Sodium	MC
Yttrium	70	Fluorine	$\sim$720
Strontium	340	Boron	70
Rubidium	33	Beryllium	6
Bromine	20	Lithium	27
Selenium	24		

*Flameless atomic absorption

MC = Major Component

Note: Any element not listed - concentration $\leq$0.2 ppm by wt
Carbon, hydrogen, nitrogen and oxygen are excluded from these analyses.



→| 10μ |←

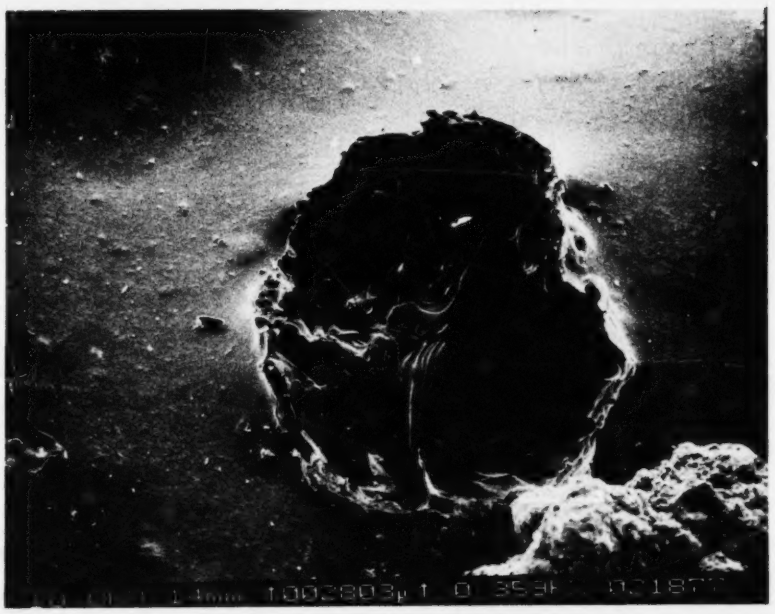


Figure 4-5. PHOTOMICROGRAPHS OF CYCLONE DUST FROM SITE A

Top - Optical Microscope: 100X

Bottom - Scanning Electron Microscope: 359X

Figure 4-5 shows a single particle under 100x magnification. The spherical section in the lower corner of the top photo is a typical fragment approximately 15μ x 50μ. Under three-dimensional observation, the fragment appeared to be 2-5μ thick. These particles were very fragile and could be easily crushed. The lower photomicrograph shows a single fragment viewed with a scanning electron microscope.

Figure 4-6 shows several other groupings of particles. Nominal particle sizes range from 20μ to 55μ. Particle fragments and hollow perforated spheres are clearly evident.

Figure 4-7 shows the particles under 20x magnification, permitting a greater field of vision and therefore including more particles. These photographs support the previous observations regarding particle shape and consistency.

Figure 4-8 shows the particulate residue which remained after the Site A tar sample was extracted with methylene chloride as described in paragraph 3.2.2. This material, which passed through the cyclone, is somewhat smaller in diameter ($d_p \approx 20\mu$) but is very similar to the material collected by the cyclone in that it consists of sintered fragments and hollow spheres. A comparison will be made of these particulates with those from three additional sources in Section 5.0 of this report.

4.4 CHARACTERIZATION OF SITE A GASIFIER ASH

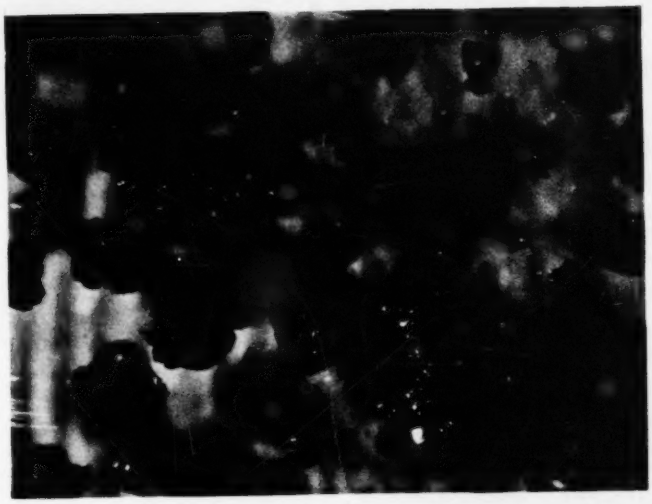
The Site A gasifier ash sample was taken from the water seal pan of the gasifier and transported as a wet aggregate. Excess water was drained off and the ash dried prior to analysis.

→| 10μ |←

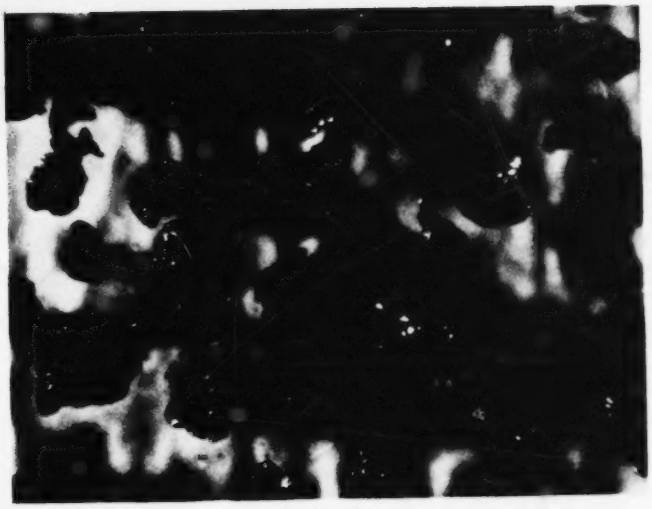
→| 10μ |←

Figure 4-6. PHOTOMICROGRAPHS OF CYCLONE DUST SHOWING SPHERICAL SEGMENTS - SITE A

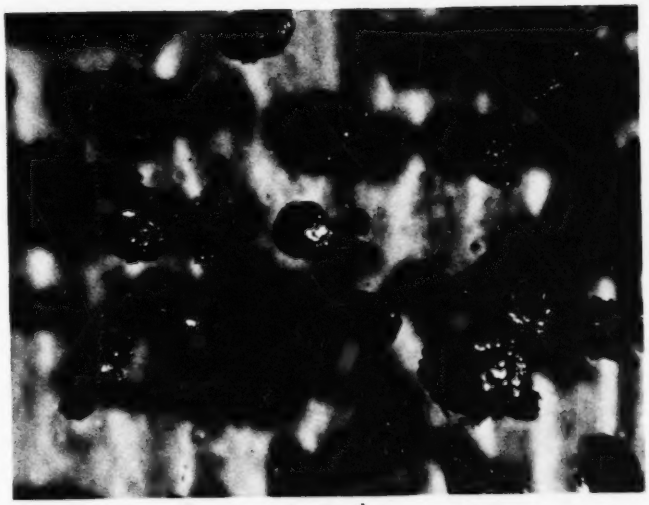
Magnification - 100X



→| 50μ |←



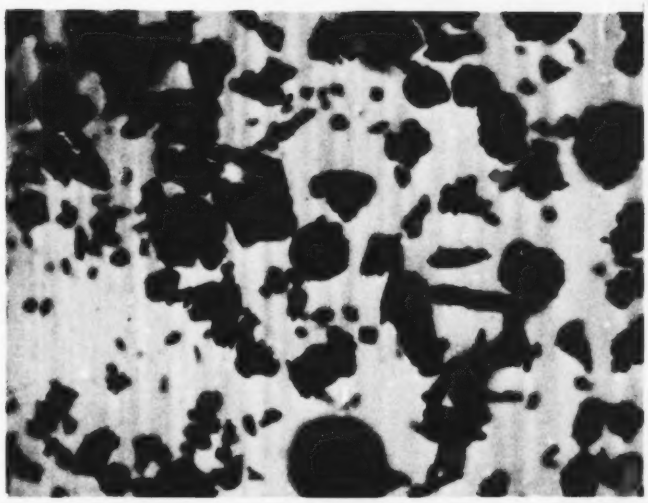
→| 50μ |←



→| 50μ |←

Figure 4-7. PHOTOMICROGRAPHS OF CYCLONE BOTTOM DUST - SITE A
Magnification - 20X

→| 10μ |←



→| 10μ |←

Figure 4-8. PARTICULATE RESIDUE FROM TAR - SITE A

(This residue passed through the cyclone and was contained in the tar sample.)

Residue = 4% by wt of tar

Magnification - 100X

4.4.1 Characterization of Organic Extract

The extraction of 50.3 grams of gasifier ash yielded 0.89 mg of organics, equivalent to 18 ppm by weight. No chromatographic separations of this extract were made. The IR spectrum of the extract showed the following: CH stretch and methyl absorption bands typical of aliphatic hydrocarbons, a split carbonyl band and a weak C-O stretch band indicative of an ester (possibly an acetate).

4.4.2 Inorganic Characterization of Trace Elements

The following table lists the trace elements found in the Site A gasifier ash sample. All values except mercury were determined by SSMS. Other elements not reported include carbon, hydrogen, nitrogen and oxygen.

TABLE 4-16. TRACE ELEMENTS IN GASIFIER ASH

Element	ppm wt.	Element	ppm wt.
Uranium	56	Niobium	82
Thorium	86	Zirconium	430
Bismuth	0.4	Yttrium	260
Lead	7	Strontium	MC
Thallium	0.5	Rubidium	120
Rhenium	0.3	Bromine	12
Tungsten	10	Selenium	20
Tantalum	2	Arsenic	4
Hafnium	10	Germanium	4
Lutetium	2	Gallium	66
Ytterbium	12	Zinc	26
Thulium	1	Copper	540
Erbium	8	Nickel	120
Holmium	11	Cobalt	61
Dysprosium	17	Iron	MC
Terbium	4	Manganese	680
Gadolinium	10	Chromium	510
Europium	5	Vanadium	MC
Samarium	28	Titanium	MC
Neodymium	56	Scandium	29
Praseodymium	42	Calcium	MC
Cerium	260	Potassium	MC
Lanthanum	280	Chlorine	230
Barium	MC	Sulfur	250
Cesium	10	Phosphorus	MC
Iodine	0.3	Silicon	MC
Antimony	1	Aluminum	MC
Tin	4	Magnesium	MC
Indium	Std	Sodium	MC
Cadmium	3	Fluorine	≈56
Silver	≤0.3	Boron	130
Molybdenum	22	Beryllium	22
		Lithium	190

MC = Major Component

Note - Any element not listed - Concentration <0.2 ppm by wt. Carbon, hydrogen, nitrogen & oxygen are excluded from these analyses.

SECTION 5.0
DISCUSSION OF RESULTS

In this section of the report selected results from Radian's analyses of Site A samples are compared with results of analyses of samples from other sources. Consideration is given to the characteristics of the particulates, trace element distributions in various samples and problems found in the characterization of organics. In addition, some limitations of the Level 1 test procedures are identified.

5.1 CYCLONE DUST FROM VARIOUS SOURCES

Comparative data for particulates from Site A as well as those from three other sources indicate properties unique to each material. The dust sources considered are identified in Table 5-1.

Table 5-1. SOURCES OF PARTICULATES

Site	Fuel	Sampling Location
(1) A	Bituminous	Cyclone Bottom
(2) A	Bituminous	Through Cyclone*
(3) B1	Anthracite	Cyclone Bottom
(4) B3	Anthracite	Through Cyclone**
(5) C1	Bituminous	Cyclone Bottom
(6) C1	Bituminous	Through Cyclone**

* Suspended in tar.

** Removed from gas duct downstream of cyclone.

( ) Numbers relate to columns in Table 5-2.

Table 5-2 shows a comparison of the particulate matter obtained from the six sources identified in Table 5-1. Attention is called to the ash content and bulk density of the cyclone bottom dust samples listed in Columns 1, 3, and 5. The large difference between the Site B sample (3) and the others is attributed to the anthracite coal used at the B Sites.

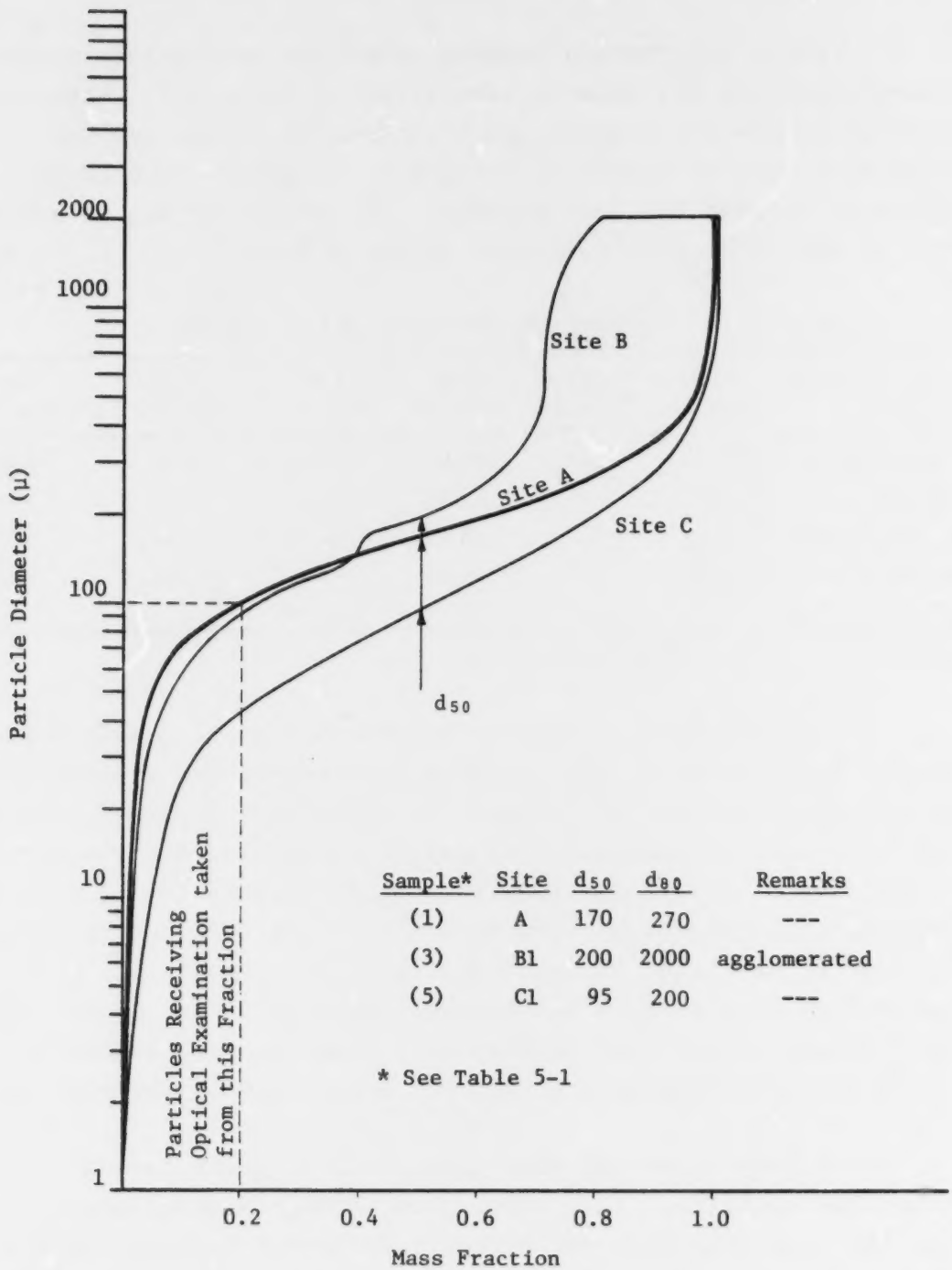
Table 5-2. COMPARISON OF PARTICULATE MATTER

Sample Location Key	(1) Site A	(2) Site A	(3) Site B1	(4) Site B3	(5) Site C1	(6) Site C1
Nominal**dp, μ	170	2-20	200.0	<1*	95.0	≈20μ
Moisture	--	--	10.5	3.3	0	--
Ash (Dry Bases)	10.19	--	47.28	54.73	15.4	10.4
Bulk Density	0.40	--	0.93	--	0.53	0.31

*Agglomerated **d_{50} (See Figure 5-1.)

The photomicrographs in Figures 5-2 and 5-3 show substantial differences in the particle geometry of the cyclone dust from the three sites. In contrast to the Site A particles which were either hollow spheres or spherical sections, both the Site B1 and the Site C1 particulates were irregular particles and were of a smaller size generally. The Site C1 cyclone appeared to bring down smaller particles using a fuel which is supposedly comparable to that used at Site A. Further, no significant amount of sphere formation was seen at Site C1. A few spheres having a dp ≈20μ were observed in the dust but these were in the minority.

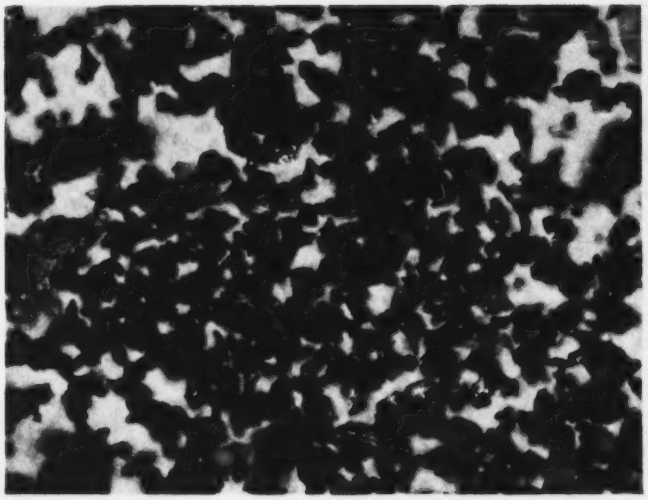
A comparison was also made of dusts which passed through the cyclones. This comparison is highly speculative since the particles were not actually collected by sampling the cyclone exit gas. Indeed, the sources were not always common to those from which bottom dusts were obtained. However, in the



Sample*	Site	d_{50}	d_{80}	Remarks
(1)	A	170	270	---
(3)	B1	200	2000	agglomerated
(5)	C1	95	200	---

* See Table 5-1

Figure 5-1. SIZE DISTRIBUTIONS OF CYCLONE DUST SAMPLES FROM SEVERAL SITES.

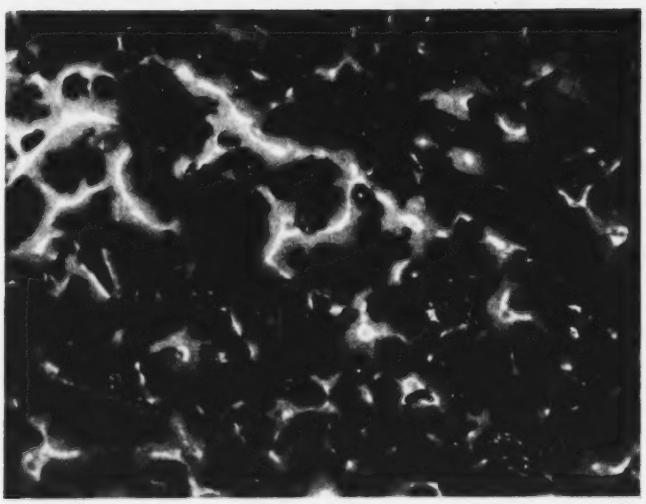


→| 10μ |←

Site B (anthracite coal)

→| 10μ |←

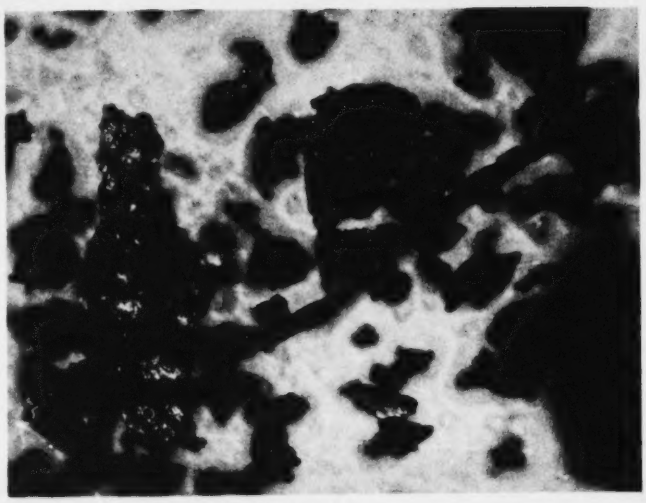
Site A (bituminous coal)



→| 10μ |←

Site C (bituminous coal)

Figure 5-2. COMPARATIVE PHOTOMICROGRAPHS OF CYCLONE DUST FROM THREE SOURCES
Magnification - 100X

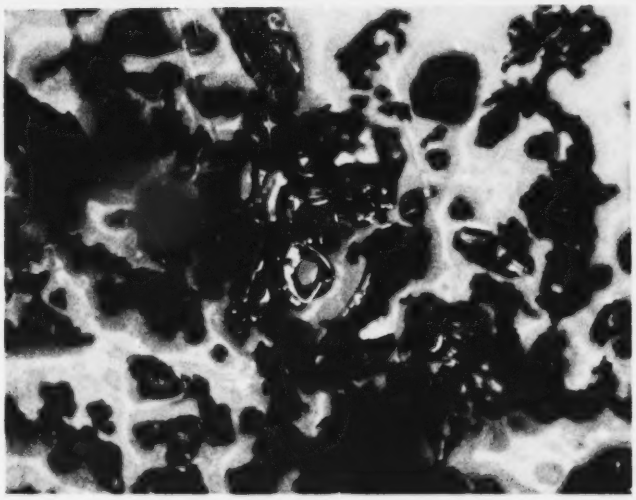


→| 10μ |←

Site B (anthracite coal)

→| 10μ |←

Site A (bituminous coal)



→| 10μ |←

Site C (bituminous coal)

Figure 5-3. COMPARATIVE PHOTOMICROGRAPHS OF CYCLONE DUST FROM THREE SOURCES
(Same as Figure 5-2 but different view of field)

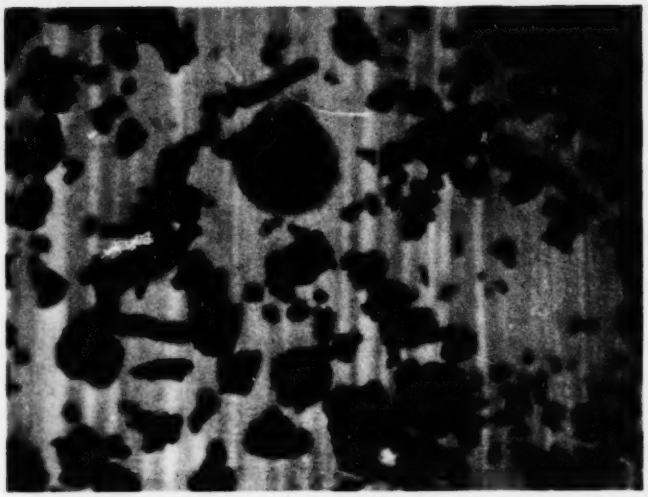
absence of more valid samples, this approach does yield some useful information.

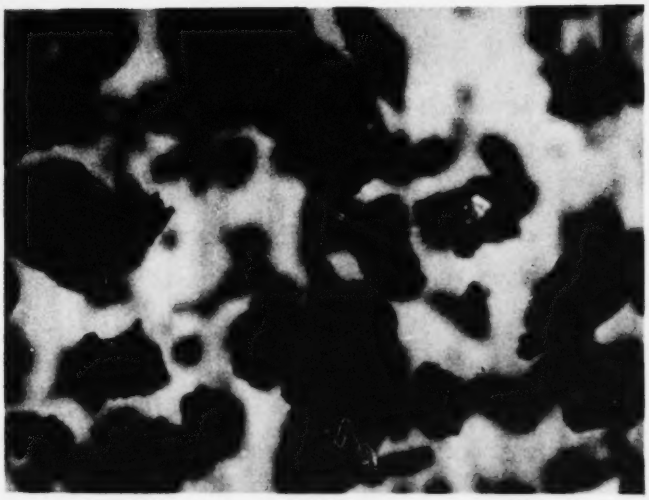
The Site A "top" dust shown in Figure 5-4 was removed from the tar sample by solution filtration. It resembled the Site A bottom ash in that it consisted mainly of spherical particles and spherical sections. Particle diameters were generally less than 20μ.

The Site B3 "top" dust shown in Figure 5-5 was taken from an accumulation of dust which was removed from the gas transport duct. It consisted primarily of agglomerates of submicron particles. It also contained translucent filaments and colored crystals, most probably due to contamination of the dust sample with mineral matter from the end use process.

Two types of "top" dust were obtained from Site C. One was scraped from the walls of the gas main downstream of the cyclone. The second was filtered from tar collected by an ESP that was used on the small process development unit. Both types of particles are very different from the Site A fines. The first Site C dust sample (See Figure 5-6) consisted of ∿10μ dp agglomerates of submicron particles. Resembling soot, it had the appearance of amorphous carbon rather than that of a sintered particle, which is characteristic of cyclone bottom dust. These particles might have been formed in the product gas duct.

The Site C tar residue shown in Figure 5-7 is truly submicron material, with the exception of some translucent crystals which appear to have grown in the tar with aging. (The sample was taken from material which had been stored on a shelf for some time.) Some crystals were "twinned," which suggests that they grew in the medium. Their composition is unknown.





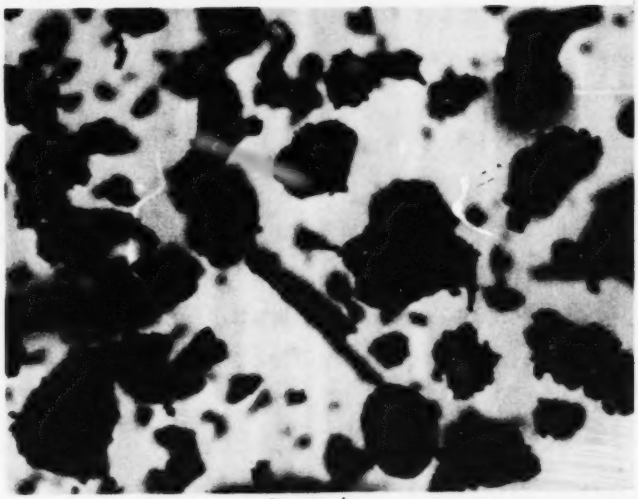
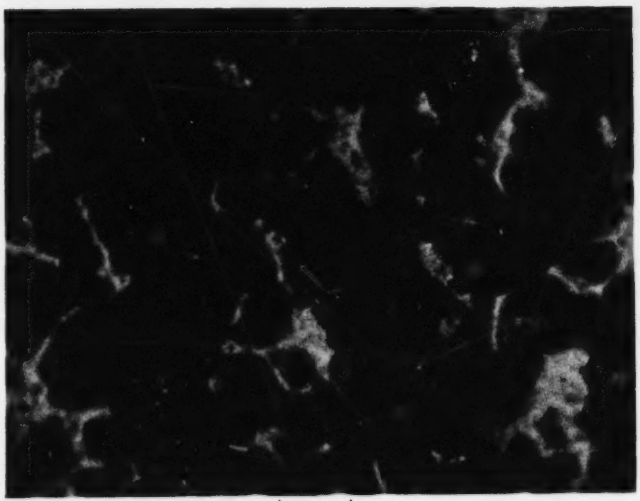
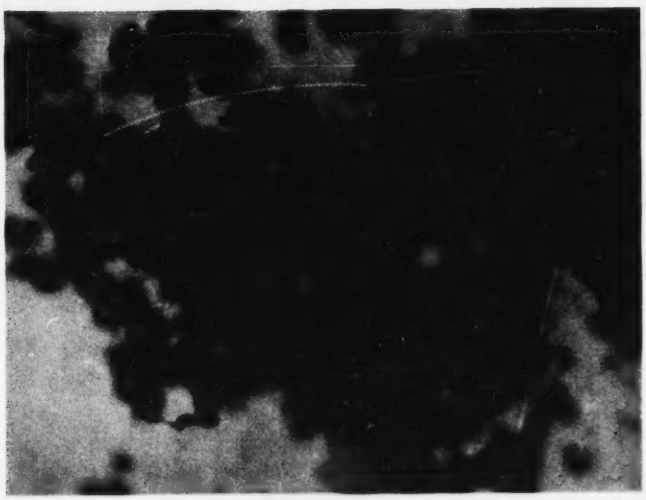


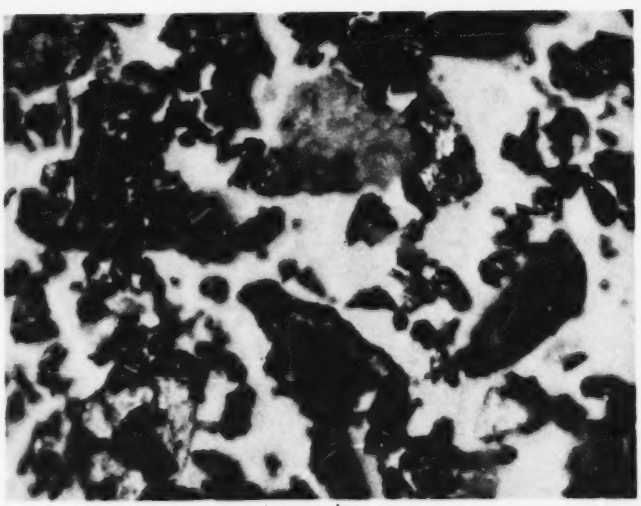
Figure 5-4. PHOTOMICROGRAPHS OF RESIDUE FROM SITE A TAR
(Particulates which passed through the cyclone)
Magnification - 100X



→| 10μ |←

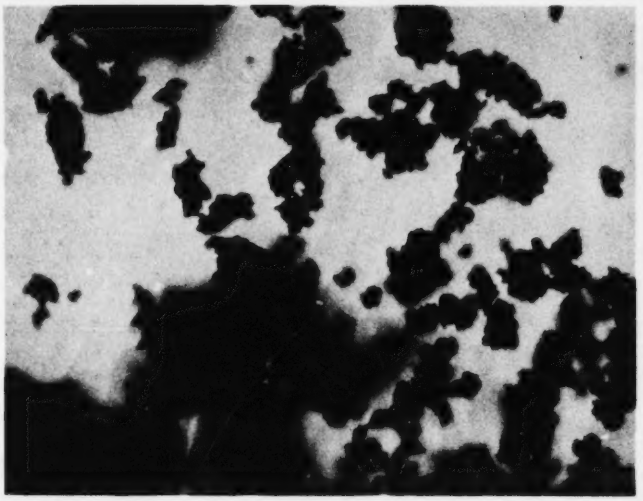


→| 10μ |←

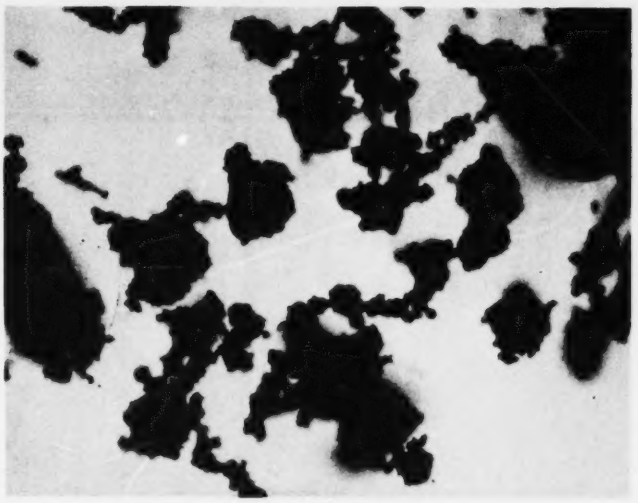


→| 10μ |←

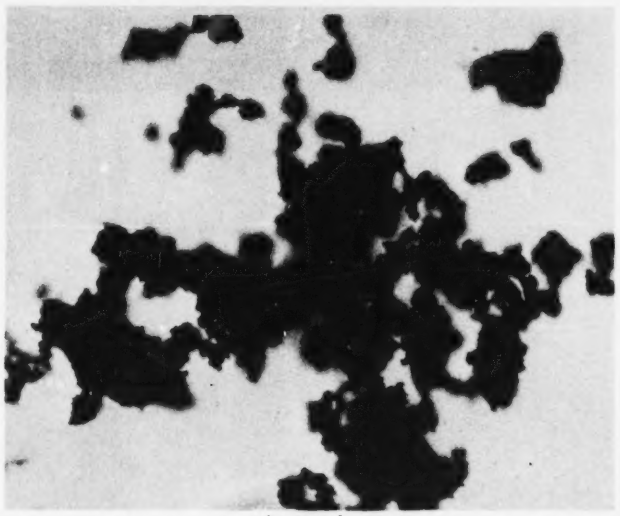
Figure 5-5. PHOTOMICROGRAPHS OF DUST WHICH PASSED THROUGH THE CYCLONE - SITE B
Magnification - 100X



→| 10μ |←



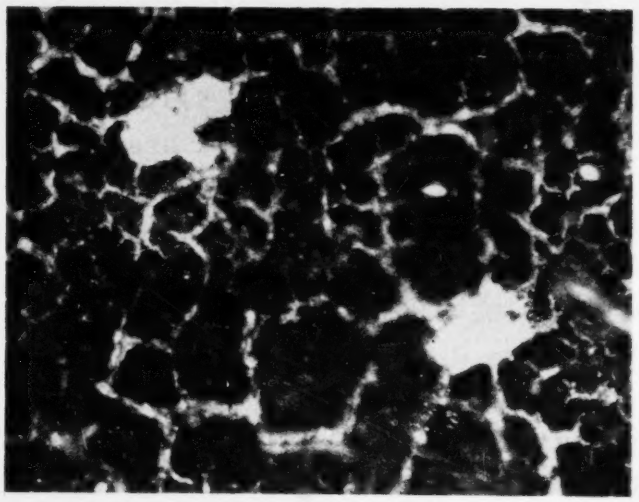
→| 10μ |←



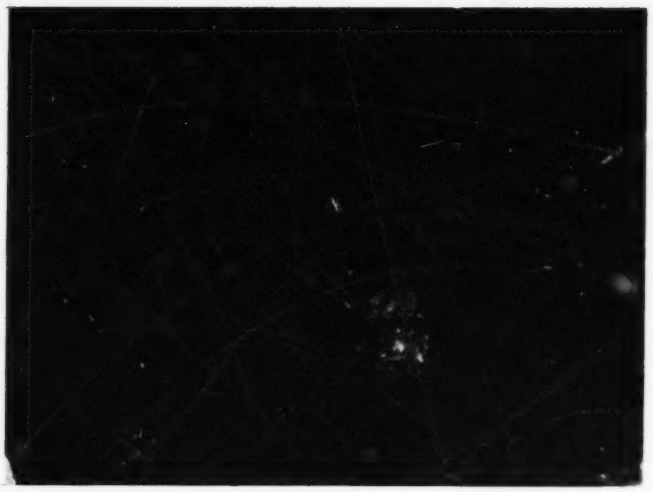
→| 10μ |←

Figure 5-6. PHOTOMICROGRAPHS OF DEPOSITS ON WALLS OF PRODUCT GAS MAIN DOWNSTREAM OF THE SITE C CYCLONE

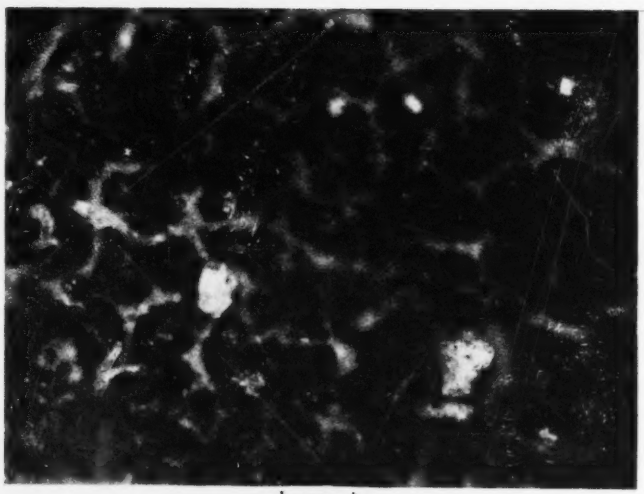
Magnification - 100X



→| 10μ |←



→| 10μ |←



→| 10μ |←

Figure 5-7. PHOTOMICROGRAPHS OF PARTICULATE RESIDUE IN TAR - SITE C

(These particles passed through the cyclone and were trapped with the tar in the electrostatic precipitator.)

Magnification - 100X

The "mud flat" pattern in the photomicrograph (Figure 5-7) is caused by the submicron particles while the light lines are ridges in the filter paper. The material contained no spheres as was evident in the Site A tar residue (see Figures 4-8 and 5-4).

These preliminary data on the particulates produced by coal gasification suggest that extreme differences in composition and physical properties of particulates can be anticipated from site to site. The differences may relate to the type of coal, system design and presumably, operating conditions.

5.2 TRACE ELEMENT LEVELS IN SAMPLES FROM SITE A

Although the sampling procedures used for this study do not justify definitive judgments regarding the transport and fate of trace elements in the process, Table 5-3 was compiled so that comparisons of the trace element levels in the respective Site A samples could be made.

5.3 COMPARISON BETWEEN QUENCH LIQUOR AND WATER QUALITY STANDARDS FOR TRACE ELEMENTS - SITE A

The levels of trace elements in the Site A quench liquor are listed in Table 5-4 along with quality standards for several types of water. The levels imposed by the standards are equaled or exceeded for nearly every element listed. The largest deviation is in the case of selenium. Its concentration in the Site A quench liquor is 400 times the quality standard for surface and public intake waters and 80 times the quality standard for irrigation water.

Table 5-3. TRACE ELEMENTS IN SAMPLES FROM SITE A

	Ash	Cyclone Bottom Dust	Liquor	Tar
Uranium	56	--	--	--
Bismuth	0.4	<2	--	--
Lead	7	60	0.04	10
Mercury	NR	0.01	0.007	0.12
Barium	MC	460	0.1	27
Antimony	1	8	0.1	0.8
Cadmium	3	<2	≤0.02	--
Molybdenum	22	14	0.06	1
Selenium	20	24	4	3
Arsenic	4	27	0.2	4
Zinc	26	85	0.07	7
Copper	540	130	0.1	3
Nickel	120	30	0.07	5
Chromium	510	90	0.03	3
Vandium	MC	100	0.004	0.8
Titanium	MC	MC	0.05	29
Chlorine	230	720	0.3	6
Sulfur	250	MC	MC	520
Fluorine	≈56	≈270	≈2	≈22
Boron	130	70	2	19
Beryllium	22	6	--	0.1
Lithium	190	27	0.2	4

All values are expressed as ppm wt. except for the liquor where values are expressed as μg/mℓ.

Table 5-4. LEVELS OF TRACE ELEMENTS IN QUENCH LIQUOR FROM SITE A VERSUS WATER QUALITY STANDARDS (μg/ℓ)

Element	Surface Water	Irrigation Water	Public Water Intake	Site A Quench Liquor
Antimony	--	--	--	0.1
Arsenic	0.05	1.0	0.1	0.2
Barium	1.0	--	--	0.1
Beryllium	--	--	--	--
Boron	1.0	0.75	1.0	2.0
Cadmium	0.01	0.005	0.01	$\leq$0.02
Chromium	0.05	5.0	0.05	0.03
Fluorine	--	--	--	2
Mercury	--	--	0.002	0.007
Lead	0.05	5.0	0.05	0.04
Manganese	0.05	2.0	--	0.03
Molybdenum	--	0.005	--	0.06
Nickel	--	0.5	--	0.07
Selenium	0.01	0.05	0.01	4
Vanadium	--	10.0	--	--
Zinc	5.0	5.0	5.0	0.07
Copper	1.0	0.2	1.0	0.1

5.4 COMPARISON OF THE ORGANIC CONSTITUENTS FOUND IN SELECTED SAMPLES

The organic constituents found in selected Site A samples, when compared with those found in supposedly similar samples from other sources, showed substantial differences in component distribution. These differences are discussed below.

5.4.1 Comparison of Site A Quench Liquor with Site C "Wash Box" Water

This comparison shows that the Site A quench liquor was carrying 3.8 times the load of organics that was being carried by the Site C wash box liquor. More surprisingly, the component distribution is strikingly different. Table 5-5 shows that the methylene chloride extraction step removed approximately 60% of the solvent extractables from both quench liquors. However, the composition of the respective extracts differed substantially as shown by the IR spectra in Figures 5-8 through 5-10.

Table 5-5. COMPARISON OF EXTRACTABLES FROM QUENCH WATER

	mg/ℓ		% of Extractant	
Extractant	Site A	Site C	Site A	Site C
$MeCl_2$	2231	411	57.7	64.6
Ether (neutral)	1457	78	37.6	12.3
Ether (acidified)	177	146	4.6	23.1
TOTAL	3865	635	100.0	100.0

The respective analyses of the methylene chloride extracts show that the Site A quench liquor contains predominantly aromatics, particularly phenols. The ether extract of the Site C wash box liquor seems to contain more aliphatic hydrocarbons

First Extraction

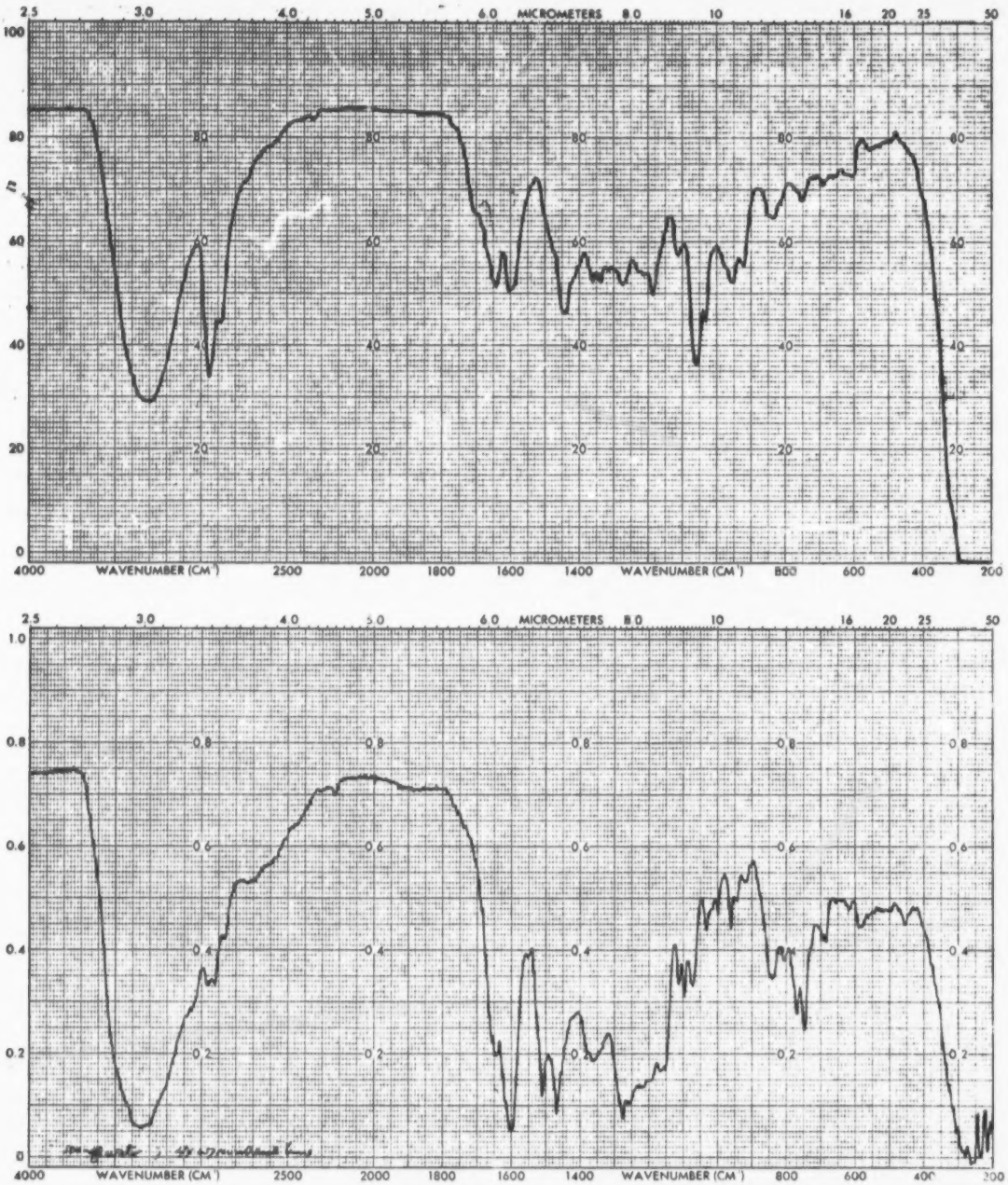


Figure 5-8. METHYLENE CHLORIDE EXTRACT OF QUENCH WATER
Top: Site C Bottom: Site A

Second Extraction

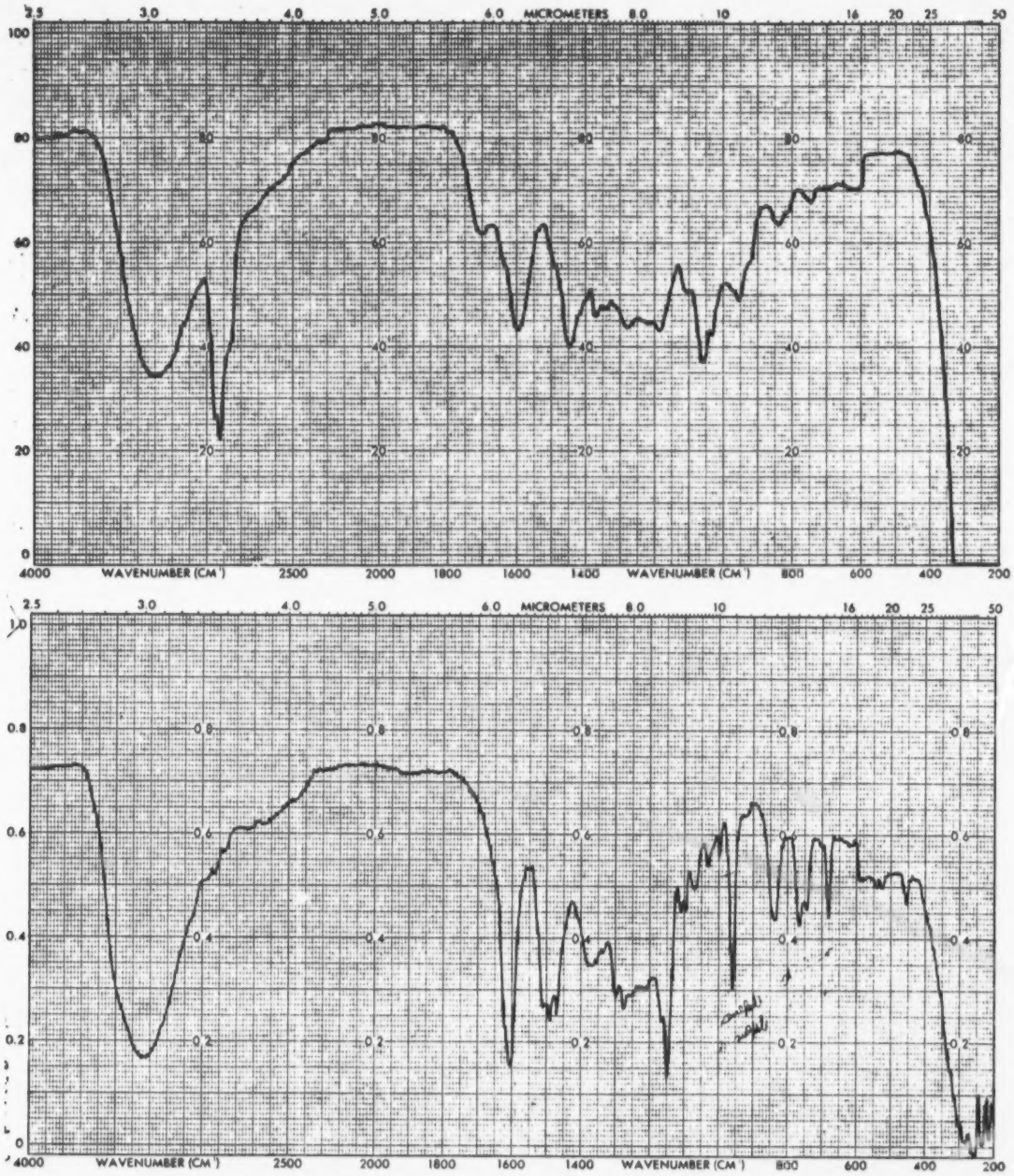


Figure 5-9. INFRARED SPECTRA OF ETHER EXTRACT OF QUENCH WATER
Top: Site C Bottom: Site A

Third Extraction

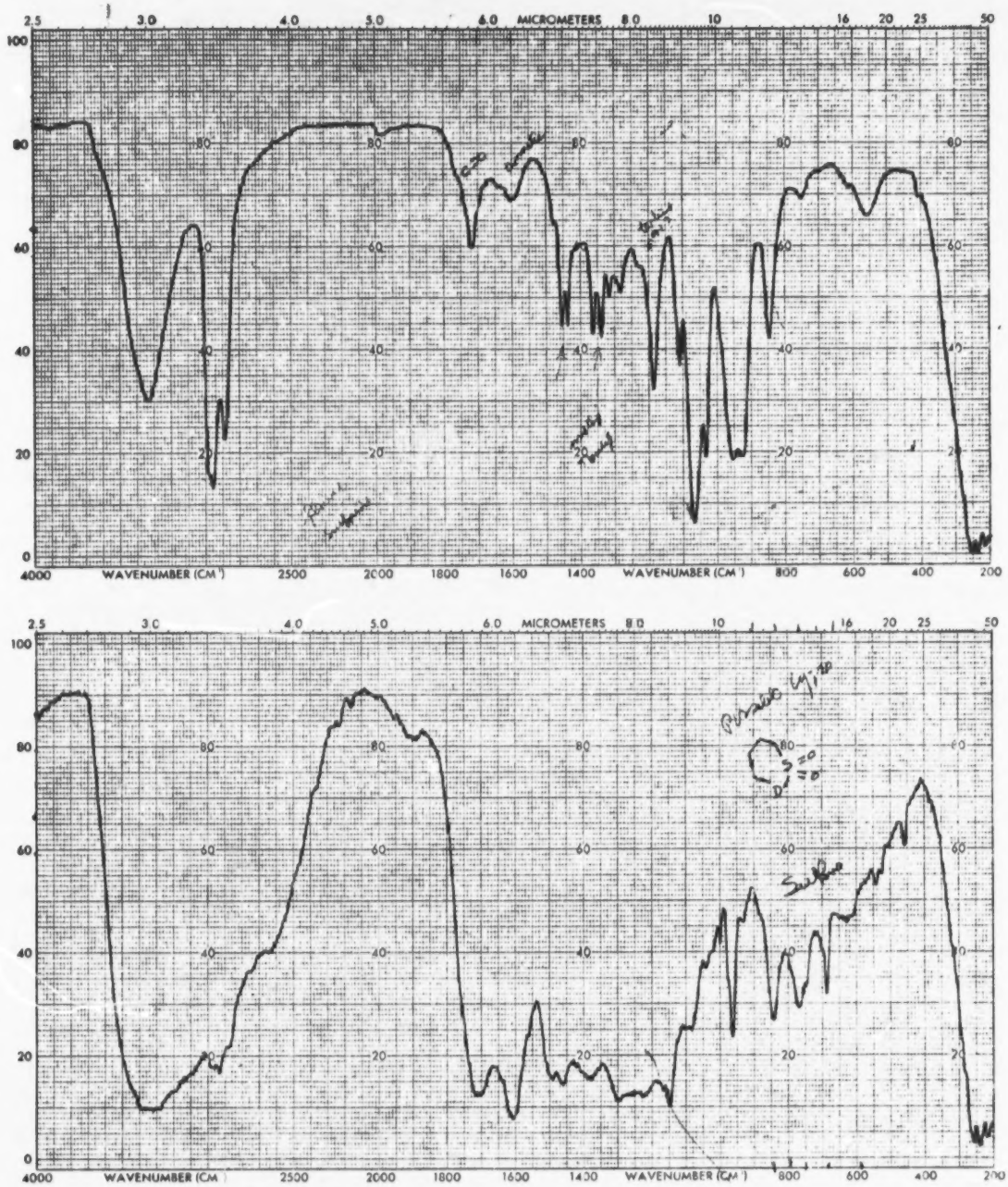


Figure 5-10. ETHER EXTRACT OF ACIDIFIED QUENCH WATER
Top: Site C Bottom: Site A

including carbonyls (1730 cm^{-1}) and some primary alcohols (1060 cm^{-1}) relative to the amount of aromatics (1600 cm^{-1}) present. Some of these effects may have been the result of the age differences in the two samples.

Both the ether and the acidified ether extracts of the Site A sample show a number of sharp bands in the fingerprint region of the spectrum (1000 to 600 cm^{-1}). These bands have not been assigned. Assignment of these fingerprint bands is, at best, subject to doubt with complex mixtures. Although sharp bands like those present in these spectra are common to substituents and polyaromatic compounds, it is difficult to believe such an assignment when it is recognized that this material was not extractable from water with methylene chloride. Since no carbonyl absorbance is present, the material cannot be an organic acid. Nor can it be an aliphatic alcohol, since no significant OH stretch is present. The absorbance is probably due to an inorganic salt or a metal organic substance, possibly a pyrosulfite. More work at a species specific level would be required to characterize this substance which is present in significant amounts. The doublet in the spectrum of the acidified ether extract of the Site C water at 950 cm^{-1} could well be due to a similar type of substance. However, the Site C extract shows a significant amount of a primary alcohol in addition to the substances present in the Site A ether extract.

5.4.2 Comparison of the Organic Composition of Tar from Site A and Site C

The Site A tar was a solid at room temperature whereas the Site C tar was a moderately viscous, free-flowing liquid. The relative mass distributions of the respective LC fractions are shown in Figure 5-11. It is evident from these graphs that

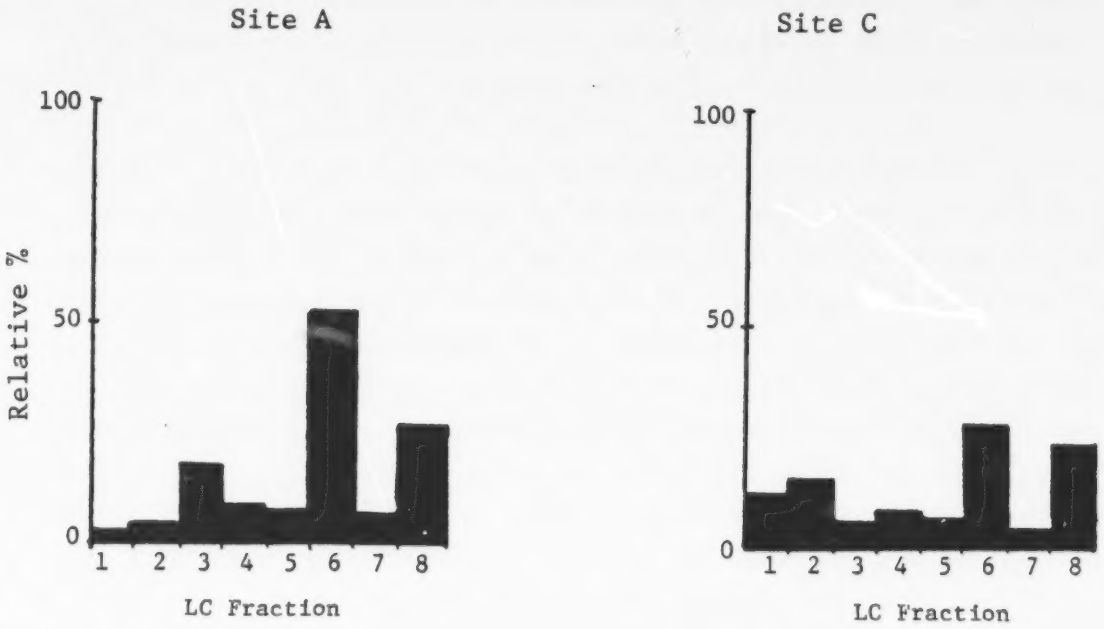


Figure 5-11. RELATIVE MASS DISTRIBUTIONS OF TAR EXTRACT FRACTIONS

the paraffinic and nonpolar aromatics are more abundant in the Site C tar. This difference in distribution may be the result of a loss of lights in the Site A quench system. The IR spectra of the respective fractions, for practical purposes, are comparable. Differences in the relative amounts of isomeric species exist but their functional compositions are similar. The major difference is seen in the IR spectra of Fraction 8. The Site A sample contains a stronger >C=O and a -C-O- vibration indicative of acetates, suggesting that the material has been in a more oxidative environment.

5.5 CONSIDERATION OF SOME LIMITATIONS OF THE LEVEL 1 TEST PROCEDURE

The Level 1 test procedure suffers limitations in two areas: the extraction procedure and the IR spectral characterization procedure. These limitations are discussed below.

5.5.1 Extraction Procedure

A modified Level 1 extraction procedure followed by two ether extractions, as described in section 3.2.1 of this report, was used on the quench liquor. The recommended Level 1 extraction procedure was modified from a 20/1 sample/extractant ratio to a 1/1 ratio in order to assure a more complete transfer of polar organics into the organic phase. This extraction was followed by ether extractions at pH 7 and pH 1 to determine whether any substances remained in the condensate following the Level 1 extraction step.

The test results, as described in section 4.1.1 indicate that four equal volume extractions with methylene chloride did not recover all of the organics present in the aqueous phase.

Therefore, a methylene chloride extraction step alone does not appear to be adequate for purposes of assessing the organics present in quench liquors from coal gasification processes. This is because a variety of substances produced during the thermal degradation of coal do not partition adequately into methylene chloride from water. The following discussion is provided to support this conclusion.

If only the mass of the extracted organic material is considered, then the results of the $MeCl_2$ extraction step may fall within the ±2 to 3 specification of the Level 1 procedure. However, when composition is included in the consideration, this might not be the case since the materials not extracted by the methylene chloride are undoubtedly chemically different from those which are. Therefore, the procedure should be modified to include the supplementary extractions.

Attention must also be called to the specified ratio of the sample to the extractant because the Level 1 procedure states: "normally three 500-mℓ methylene chloride extractions of 10-liter samples should be sufficient" (Reference 1, Section 8.3.1, page 97). This recommended ratio may lead to seriously misleading results since the recommended 1:20 volume ratio could leave many components in the water, as is demonstrated by the following relative mass transfer equation:

$$F = \frac{KV_e}{V_s + KV_e}$$

where,

$$K = \frac{[\text{Solute}]_{\text{extractant}}}{[\text{Solute}]_{\text{sample}}} = \text{Partition coefficient}$$

F = relative mass transfer per extraction

V_e = mℓ volume of extractant ($MeCl_2$), and

V_s = mℓ volume of sample (liquor).

To comply with Level 1 specifications of ±2 to 3, on the order of 50% of the mass of a given component must be transferred in three extractions. In order to transfer 50% of the dissolved mass in three extractions, about 20% per extraction must be transferred. Using this F value in the mass transfer equation with a volume ratio of 1:20 as specified, a K = 5.0 is obtained. This calculation shows that any substance with a partition coefficient significantly less than 5.0 would fall outside the specification of the method. Because of the wide range of polarities shown by the substances produced in coal gasification processes, it is recommended that as a minimum, a four-step extraction with a total volume equaling the sample volume be used for all batchwise extractions. It is further recommended that an extraction at pH 1 be used following extraction at pH 7 to effect a more complete transfer of polar organics for analysis. A reasonable alternative is to use a commercial continuous extractor which can be run overnight, providing a large number of transfers virtually unattended.

5.5.2 IR Spectral Characterization

The IR spectral characterizations of these grab samples have provided information that is useful to the planning of a more detailed sampling program. The functional groups that were identified, indicate the types of substances that are present. When this information is used in combination with the LC data, it provides a profile of the substances that must be dealt with

in a detailed sampling program as well as in a Level II analysis to obtain species specific data. Indeed, considerable information is contained in the spectra that is useful to the analyst. However, taken alone, this information is inconclusive and must be substantiated by additional analysis.

One area where the roll of IR is limited is with materials such as tars which we know to be complex organic materials. Spectral characterization of complex mixtures such as tars using Level 1 procedures is of limited value to an environmental assessment since their spectra are so diffuse and devoid of definitive bands. The intermolecular bonding in tar restricts molecular vibration and therefore inhibits the generation of sharp bands by which definitive assignments can be made. As a consequence, no distinct fingerprint exists to identify tar as an emission from a suspected source. Further, without the use of more sophisticated separation steps, specific hazardous species cannot be identified. The Level 1 technique can be used only to compare tars from different sources and to show that they are probably similar in composition. Stated more broadly, when the spectrum of one tar is identifiably different from that of another, it can be asserted that the tars are different. However, when the spectra of two materials are similar, but not identical, it can only be asserted that the materials are probably similar on a bulk basis. Indeed, either substance could contain harmful amounts of hazardous species not indicated by the spectra. If such a substance were detected by other means, the IR spectrum could not be used to establish that the tar was the source.

In order to discriminate between various types of compounds having similar functional groups, it is generally necessary to rely on the fingerprint region of the IR spectrum

(1430-800 cm^{-1}) since absorbance in that region tends to be molecular specific. Therefore, to discriminate between polycyclic and alkylaromatic hydrocarbons, spectral bands in the fingerprint region must be used. However, unless the sample is in solution and relatively free of interferences from other compounds, band assignments tend to be ambiguous. Spectral bands can change position in the fingerprint region depending on the physical state of the component in the sample as well as on the nature and position of substituents in the molecule. These uncertainties limit the utility of the fingerprint region as a tool for functional group discrimination in complex mixtures. Assignments must therefore be based on group frequencies which are relatively nondefinitive and indeed of limited value in detecting polycyclic aromatic compounds. Consequently, consideration of an alternative screening method is warranted.

Ultraviolet spectrometry is considerably more sensitive than IR spectrometry to polycyclic aromatic hydrocarbons and is capable of detecting traces of multiring compounds. The UV technique should be included in the screening procedure as a method of detecting traces of polycyclics in samples and chromatographic fractions. Since an increase in ring number causes the UV absorption maxima to shift to a longer wave length, the technique can be useful in characterizing polyaromatic materials. For example, a UV absorbance at a wavelength greater than 330 nm can be taken as an across the board indication that polycyclic substances are potentially present, particularly if that absorbance is observed in LC fractions 2 or 3. Further, the UV technique is much more sensitive than IR.

SECTION 6.0
SUMMARY OF CONCLUSIONS AND RECOMMENDATIONS

Several judgments can be made regarding the data obtained and the methods used in this study that will affect the low-Btu data acquisition program. These judgments are expressed in the conclusions and recommendations listed below:

Organic Constituents in Site A Quench Liquor:

- The major organic constituent of Site A quench liquor was phenol with lesser amounts of di- and trisubstituted components; volatile organics fell mainly in a chromatographic boiling range of 160-200°C.

Ether Extracts:

- The ether extracts of the Site A quench liquor differed in composition from the Site C ether extract. The Site C extract appeared to contain more aliphatic material than the site A extract. Active functional groups such as C-H, $\rangle$C=O, and -C-O were not as evident in the spectra of the Site A ether extracts as they were in the Site C sample.

- Only 57% of the extractable organics were removed from the Site A quench liquor by the Level 1 procedure. The remaining organics were removed by supplemental extraction with

ether from a neutral, then acidified sample. The spectral compositions of the respective extracts were different.

Trace Element Levels:

- The levels of trace elements in the Site A quench liquor equaled or exceeded the levels listed in the Federal Water Quality Standards for nearly every element. The largest deviation was shown by selenium at 4 ppm (400 times the quality standard for surface water).

Cyclone Dust:

- The Site A cyclone bottom dust appears to consist of hollow spheres with a nominal particle diameter (d_{50}) of 170μ. Fines appear to be sections of broken spheres. The Site C dust is smaller and irregular in shape, but of comparable bulk density. The Site B dust (anthracite coal) has a particle shape comparable to the Site C dust but is more than twice as dense and was highly agglomerated. Particulates extracted from the Site A tar had the same physical characteristics as the bottom dust, though smaller in diameter.

Hypothesis:

- These observations indicate that the characteristics of the particles in the gasifier

exit gas may depend on fuel types, system design, and/or operating conditions. Since those particulates may provide a mechanism for trace element transport, particle removal must be studied over a wide range of conditions to obtain information needed for a comprehensive and reliable assessment of the fate of trace elements in gasification processes.

Procedures:

- The Level 1 procedures make virtually no provisions for detecting organic sulfur or nitrogen compounds. The IR spectral procedure is ideal for detecting aromatic, carbonyls and alcohols, but it is not well suited to detecting C-S-, and C-N. Substances that contain hydrogen-bonded hydroxl groups produce such broad bands that discriminating spectral assignments are virtually impossible. Conversely an assignment that includes all possible structures that might be represented by a broad band is apt to be so inclusive that it is not informative. A limited amount of additional work, to detect the presence of organic sulfur and/or nitrogen would be of value to a Level 1 assessment. At a minimum, an elemental analysis for S and N in the eight LC fractions could be included as a valuable "add on" to a Level 1 test.

Additional Needs - HPLC:

- A moderate amount of high-pressure liquid chromatography (HPLC) or at least thin layer chromatography could be a valuable adjunct to the Level 1 procedures when used on major fractions (for example, Fraction 6 in this study) to discriminate between the nonvolatile component classes. Such additional work can provide valuable information for both the Level 1 assessment and the Level II test plan. HPLC is needed to characterize these polar materials since they do not lend themselves to characterization by GCMS. Further, since they are soluble in water, they are of environmental concern.

Additional Needs - UV Spectrometry:

- UV or Fluorescense spectrometry should be used to detect polycyclic aromatics in the LC fractions. These techniques are several orders of magnitude more sensitive to polycyclics than IR and they can be used in conjunction with TLC for additional characterization as required. Since sample preparation is very simple, this work could be done at minimal additional cost to a basic Level 1 program.

REFERENCE

1. Hamersma, J. W., S. L. Reynolds and R. F. Maddalone, IERL-RTP Procedures Manual: Level 1 Environmental Assessment. EPA-600/2-76-160a, EPA Contract No. 68-02-1412, Task 18. Redondo Beach, CA, TRW Systems Group, June 1976.